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Assessment of Fluidized-Bed Combustion Solid Wastes for Land Disposal. Volume 4 Appendix F

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ASSESSMENT OF FLUIDIZED-BED COMBUSTION SOLID WASTES FOR LAND DISPOSAL

Volume 4. Appendix F

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a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field Group
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Waste Disposal	Stationary Sources	14B
Combustion		07A, 07D
Fluidized Bed		21B
Processing		13C
Coal		11G
Assessments		13H
Desulfurization		21D
Earth Fills		
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Abstract

The report, including 10 appendices, gives results of: (1) an analysis of representative samples of fluidized-bed combustion (FBC) wastes, (2) an assessment of the characteristics of leachates generated from the wastes under laboratory and field (landfill) conditions, (3) a characterization of the attenuation of the leachates by earth materials that are typical of disposal settings, (4) development of a way to predict the leachate generation behavior of FBC wastes under landfill conditions on the basis of laboratory test results by establishing a rigorous statistical relationship between the laboratory and field leaching results, and (5) assessment of the compatibility of commonly used landfill liner materials with FBC waste leachates. FBC is an emerging energy technology that holds promise for both high efficiency of energy conversion and minimization of adverse air quality impacts. A major advantage of FBC is that high-sulfur coal can be burned without the use of flue gas desulfurization equipment to meet air quality standards. The solid residues that are generated in an FBC unit are usually larger in volume and have different properties than the typical bottom ash from a conventional boiler.

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COMPATIBILITY OF LANDFILL LINER MATERIALS WITH FBC WASTE LEACHATE

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NOTE: In some cases, lengthy tables and large groups of figures in this appendix are removed from the text and included as attachments to minimize interruption of the flow of the text.

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F.1 Overview

In the event that some FBC residues are classified under the Resource Conservation and Recovery Act as hazardous wastes, these residues will require secure (leak-proof) disposal sites. When FBC residues are classified as nonhazardous, leakage of leachate from the disposal site will be permitted so long as no underground drinking water source beyond the solid waste boundary is contaminated. Inasmuch as the bulk of FBC wastes will likely be classed as nonhazardous (see Appendix D and Reference F-1), the emphasis of this program is on the generation of leachate and the subsequent interaction of the leachate with the substrate at disposal sites. However, studies of the effect of FBC wastes on landfill liner materials that could be used to prevent leakage were included in the program to account for those future FBC wastes that may be classified as hazardous. Some future nonhazardous FBC waste landfills may also have liners incorporated in their design.

This appendix presents the results of an investigation of the chemical compatibility and the stability characteristics of six synthetic liner materials and one clay when exposed to the leachate from fluidized combustion residues. The leachate used in the study was derived from protocol Step 1 leaching of Exxon PFBC waste. The results set forth here are useful where an upper liner is being considered for a moisture barrier above the waste body or a bottom liner is being considered to inhibit downward moisture (leachate) movement.

The liners selected for investigation are as follows:

- Neoprene;
- Polyvinyl chloride (PVC);

- Chlorinated polyethylene (CPE);
- Chlorosulfonated polyethylene (Hypalon);
- Butyl rubber;
- Ethylene propylene diene monomer (EPDM) rubber; and
- Sodium bentonite.

The six organic-based liners were placed in continuous contact with the leachate from Exxon PFBC waste. The liner integrity was studied by monitoring three parameters: 1) Tensile strength of the liner (ASTM method D412); 2) Liner surface characteristics as determined by scanning electron microscope (SEM) observations; and 3) Total organic carbon (TOC) concentration in the leachate contacting the liner. Tensile strength and liner surface characteristics were also determined for each liner material prior to contact with leachate. Enough strips of each liner were placed in contact with leachate to allow three per month (for 18 months) to be removed for measurements of tensile strength (in triplicate) and for SEM observation.

The sodium-exchanged Wyoming bentonite was placed in continuous and intimate contact with leachate from Exxon PFBC wastes, and the following observations were made to assess clay lattice degradation:

- Crystalline structure by X-ray powder diffraction;
- Physical observation by SEM; and
- Aluminum concentration in the leachate.

Each month aliquots of both the clay mineral and the leachate were taken, and the above parameters determined. SEM and X-ray diffraction allowed direct observation of changes in the clay mineral structure, whereas an increase of aluminum concentration in the leachate was used to indicate if chemical attack by the clay lattice by the leachate had occurred.

Although a gradual loss in tensile strength of the synthetic liners was observed over eighteen months of testing, tensile strength generally remained high. Strength retention was as high as 80% (for PVC) and as low as 60% (for CPE and neoprene). Hypalon (1590 psi to $\geq$2130 psi) and PVC (1300

psi to 1630 psi) had the highest tensile strengths, whereas neoprene (830 psi to 1420 psi) and CPE (630 psi to 1430 psi) had the lowest strengths. CPE swelled 31% on a mass basis over the eighteen months of exposure to the leachate, whereas most of the other liners swelled less than 10%.

The total organic carbon concentrations of the FBC leachate solution increased slightly with exposure time for all liner materials except neoprene. The greatest losses of TOC to the leachate were observed for CPE (as high as 10.2 mg TOC/g CPE) and Hypalon (as high as 10 mg TOC/g Hypalon) for any single month. Neoprene and EPDM rubber exhibited the smallest losses with concentrations up to only to 0.35 mg TOC/g neoprene and 0.64 mg TOC/g EPDM rubber for any month. Losses in TOC and tensile strength were not directly related.

Development of crystalline calcium compounds on all of the synthetic liners partially to fully obstructed viewing of the polymer surfaces after interaction with PFBC leachate. CPE and Hypalon showed some signs of degradation in visual appearance. Neoprene appeared to undergo the least visual degradation. The surfaces of PVC, butyl rubber, and EPDM rubber were too encrusted with crystalline calcium compounds to observe the polymer surfaces after interaction with PFBC waste leachate.

The overall conclusions from the results of the analyses performed on the six polymer liners is that these liners appear to be fairly stable under accelerated interaction with PFBC leachate. The following conclusions were drawn from individual test components:

- The tensile strength results indicate that the Hypalon (0.03 inches thick) and PVC (0.03 inches thick) provide the best landfill liner resistance when exposed to PFBC leachate, whereas the neoprene (0.06 inches thick) and CPE (0.03 inches thick) were the least stable polymers tested. However, since none of the liners showed rapid breakdown under accelerated leaching conditions, they should all provide good protection against seepage of FBC waste leachate.

- Total organic carbon analysis of the leachate showed that neoprene and EPDM rubber have the greatest long-term integrity, whereas Hypalon and CPE exhibited the lest integrity. However, none of these concentrations was exceptionally high.

- SEM examinations of the polymers did not clearly define surface degradation because of surface obstruction from calcium compounds precipitated from the FBC waste leachate. While neoprene appeared to be the least affected by the leachate interaction, CPE and Hypalon showed some gradual surface erosion. The surfaces of PVC, butyl rubber, and EPDM rubber were too encrusted to examine, and no definitive conclusions can be drawn about visible degradation of their surfaces.

In the bentonite clay tested, the original montmorillonite was a mixed layer clay having two slightly different structural units, as shown by a double peak with a less intense broad peak at around 61° 2θ and a more intense peak at 62° 2θ. The sodium-exchanged montmorillonite changed to a calcium-substituted clay during the first month of interaction as indicated by the shift in the 001 line from 11 Å to 15.5 Å and changed in position, intensity, and half-widths in the higher order 001 lines. After one month of interaction with PFBC leachate, a new crystalline phase (ettringite, calcium aluminum sulfate silicate carbonate hydroxide hydrate) began to form and gradually increased with exposure time to PFBC waste leachate.

The original montmorillonite contained two separate morphologies--a rosette formation and smooth large aggregates. The smooth aggregates contained less magnesium and more aluminum than the rosettes; both contained sodium, silicon, a little iron, and no appreciable calcium concentration. Although no morphological changes in the clay were visibly apparent, after one month of interaction with FBC leachate, the XRF scans indicate the loss of sodium and replacement by calcium. Crystalline phases not present in the original specimen began to form adjacent to montmorillonite aggregates after

the first month of interaction. These crystalline phases may have been calcium sulfate, calcium carbonate, calcium oxide, ettringite, or other complex silicates. Over 18 months of interaction with FBC leachate, no appreciable differences occurred in the visual appearance of the clay aggregates, although the concentration of new crystal-line formation increased slightly from the first to the eighteenth month of exposure.

No aluminum concentration in the FBC leachate suspension with the montmorillonite ever exceeded the initial background aluminum concentration of the PFBC waste leachate, which was 0.614 mg/L.

The experimental results from the tests applied to the clay liner indicated that the montmorillonite generally exhibits stable physical properties and is chemically suitable as a candidate liner for FBC waste leachate. The following conclusions were drawn from individual test components:

- The X-ray diffraction analyses of the montmorillonite showed that the montmorillonite structure remained unchanged with FBC leachate interaction, with the exception of the formation of ettringite. It is possible that the ettringite formed from a reaction of clay and FBC leachate components.

- The SEM analysis of the clay confirmed the long-term integrity of the clay liner. No visible changes were observed in the particle morphology. The SEM analysis also confirmed, however, the slow formation of new crystalline phases. These phases were identified by X-ray fluorescence analyses as calcium compounds, probably $CaSO_4$, CaO, $CaCO_3$, and/or complex calcium silicates.

- The aluminum concentration data indicated that montmorillonite did not change in the aluminum-oxygen network of the clay over 18 months of accelerated interaction with FBC waste leachate.

Although this study found little evidence of adverse effects on the liners tested, additional laboratory testing should be undertaken for future liner applications at FBC disposal sites. Prediction of actual field behavior of these materials should include laboratory testing of puncture resistance of the polymers, permeability of all the liners, and testing in waste column arrangements. Further testing should also include complete monitoring of changes in the liner dimensions with exposure as well as chemical changes in polymer (and clay) composition. Changes in hardness should be monitored for polymer liners. These tests should also define interaction under laboratory conditions that simulate actual conditions, complete with weathering and seam testing. The clay should be tested for changes in cation exchange capacity, swelling, permeability, conductivity, and pH. A field liner study compared to laboratory study would also provide invaluable information.

F.2 Introduction

For wastes classified as hazardous under provisions of the Resource Conservation and Recovery Act of 1976 (RCRA), present regulations promulgated by the Environmental Protection Agency will require containment of leachate rather than merely attenuation. Furthermore, nonhazardous waste landfills will be required to use liners in certain circumstances. Therefore, this study was designed to measure the chemical integrity (compatibility) of candidate liners in continuous and intimate contact with FBC leachate. The long-term integrity was investigated to the maximum extent possible, subject to time (18 months) and budgetary constraints. Susceptibility of liner materials to chemical attack by leachate was the key consideration, since any satisfactory liner material is assumed to be essentially impermeable to leachates unless degraded in some manner.

The liner materials studied were;

- Neoprene;
- Polyvinyl chloride (PVC);
- Chlorinated polyethylene (CPE);

- Chlorosulfonated polyethylene (Hypalon, fiber mesh reinforced);
- Butyl rubber;
- Ethylene propylene diene monomer (EPDM) rubber; and
- Sodium bentonite (montmorillonite).

These liners were chosen because they are commonly used and they have been proposed as sanitary landfill liner materials in several prior studies of liner compatibility (Reference F-2). They have been extensively studied for other types of wastes (industrial, utility, and others), and their relative merits for application have been analyzed in terms of cost, engineering, and availability. Therefore, the focus of attention was to ascertain if any of them are chemically degraded when in contact with FBC waste leachate.

Historically, synthetic liners have provided industry with an economical method of fluid waste containment with minimal permeation. They are considered to be relatively simple to install and maintain. Their major disadvantages are potential breakdown due to chemical interaction with contained wastes, physical changes (cracking, stretching) with temperature change, ultraviolet and ozone deterioration, and their susceptibility to laceration and punctures.

Synthetic liners are vulcanizable or thermoplastic rubbers or plastics. They may be polar or nonpolar chemically, and they range from amorphous to crystalline structurally. The synthetic liners are available in a wide range of compositions, grades, and price due to manufacturer's treatment to specification, such as addition of plasticizer or addition of degradation retardant.

Neoprene is a chlorine-bearing synthetic rubber which is generally very resistant to chemical attack by oil, to weathering, to exposure to ozone, and to ultraviolet radiation. It is very similar to natural rubber in its flexibility and strength, and it is resistant to puncture or mechanical damage.

Polyvinyl chloride (PVC) has been the most frequently used liner in existing landfill facilities where synthetic liners are used. Its popularity is probably linked to its resistance to petroleum products. Unfortunately, the plasticizers used to give PVC its flexibility have been shown to be biodegradable and partially water soluble. PVC also has the disadvantage of stiffening at low temperatures; however, it is quite puncture-resistant and easy to splice.

Chlorinated polyethylene (CPE) is a completely saturated polymer, and its lack of double bonds makes it ozone-resistant. CPE resists cracking at low temperatures and is known for its good tensile strength. Its major disadvantage is its susceptibility to chemical attack by acids, caustics, and oils.

Chlorosulfonated polyethylene (Hypalon) is a synthetic rubber. It is puncture-resistant and performs well over a broad temperature range. It is not susceptible to attack by weathering, aging, oil, bacteria, caustics, or acids. Although this material is widely used, it is very expensive.

Butyl rubber is a copolymer of isobutylene (97%) and isoprene. The major advantage of butyl rubber is its ability to resist permeation and swelling in water. It is, however, susceptible to ozone deterioration and chemical attack by hydrocarbons. Splices of butyl rubber also require the use of cold-curing adhesives, which is a difficult process.

Ethylene Propylene Diene Monomer (EPDM) rubber is also a synthetic rubber. Like butyl rubber, EPDM rubber is resistant to permeation and swelling in water. Unlike butyl rubber, it resists ozone attack, aging, weathering, and most chemical attack. This difference is a result of the unsaturation in the side chain rather than the main chain, while the diene monomer introduces double bond sites for vulcanization of the rubber. EPDM rubber is not resistant to petroleum solvents, and to aromatic or halogenated solvents.

Two liner thicknesses were used. The PVC, CPE, and Hypalon (reinforced with large-gauge cord) were approximately 0.03 inches thick, whereas the rubbery polymers (neoprene, butyl rubber, and EPDM rubber) were approximately 0.06 inches thick. The choice of liner thickness depended upon availability of the liner materials.

An alternative to waste containment by polymer liners is to use naturally occurring clay. A sodium-exchanged Wyoming bentonite was tested in this study. This bentonite (a montmorillonite clay) has been used as a liner and is proposed here because it forms an impermeable layer when hydrated due to its high-swelling properties. Refined montomorillonite is commercially available for use as a liner or as an admixture to be used in conjuction with the site soil (References F-3 and F-4). Bentonite is usually composed of montmorillonite and traces of feldspar, gypsum, calcium carbonate, quarts, and other minerals. Tests have shown that bentonite can sorb five times its weight and can swell twelve to fifteen times its dry bulk volume at maximum saturation (Reference F-5). The clay also has a very high cation exchange capacity. The clay occurs naturally in Wyoming, Montana, Utah, California, and South Dakota.

The major drawback of bentonite liners is their susceptibility to attack by ionic salts which can reduce the swelling properties of the clay, causing cracks and resulting in waste fluid seepage. Prehydration with fresh water is therefore mandatory when bentonite is used. The deterioration due to saline attack has been retarded in some cases by addition of a polymer to the clay (e.g., Volclay, see Reference F-3).

F.3 Previous Liner Investigations

In 1976 the Environmental Protection Agency reported the results of the first year of physical tests and analyses of polymeric materials tested as liners for sanitary landfills (Reference F-6). After one year of exposure to simulated municipal landfill leachate, the polymeric liners did not exhibit losses in impermeability; however, some of the polymeric membranes did

swell and lose seam strength. Some of the results from that study are shown in Table F-1.

The crystalline-type polymers (polyethylene, polypropylene, and polybutylene) showed the least change. Unfortunately, the crystalline polymers are the type most prone to puncture and tear and are generally difficult to handle in the field. The thermoplastic membranes (CPE, Hypalon, PVC) tended to swell the most. The vulcanized rubbery materials (butyl and EPDM rubber) changed little during the exposure period but had the lowest initial seam strength.

It should be noted that the chemical quality of the leachate used in these immersion tests was not the same as FBC waste leachate. These tests were performed with leachate produced from refuse yielding organic acids (i.e., acetic, propionic, isobutyric, and butyric acids) not present in the FBC waste leachate. These acids have a large impact on liners that are susceptible to attack by organic acids.

In 1979 the EPA reported the results from the continuation of this study (Reference F-7). This report generally confirmed the earlier results. Specimens of CPE, Hypalon, EPDM rubber, and neoprene liners showed the greatest swelling of the twenty-eight polymers tested, as shown in Table F-2.

In the final report on lining of waste impoundment and disposal facilities, polymer liners were evaluated for compatibility with municipal waste leachate after eighteen months of exposure (Reference F-8). The final results indicated that the leachate swelled the liners more than water and that exposure from two sides yielded higher swelling values. Some of the swelling of tested materials leveled off with time, and the concentration of organic constituents declined in the leachate. Figures F-1 and F-2 indicate the swelling properties and tensile strength retention reported for this study.

TABLE F-1. EFFECT ON THE PROPERTIES OF POLYMERIC LINERS OF ONE YEAR OF EXPOSURE TO LEACHATE FROM SIMULATED SANITARY LANDFILLS (DATE IN U.S. CUSTOMARY UNITS)

Item	Exposure Time, Years	Polyethylene	PVC	Butyl Rubber	Hypalon	EPPM Rubber	CPE
Liner No.	-	21	17	7	6	16	12
Generator No.	-	19	20	21	22	23	24
Thickness, mils	0	11-12	20-21	61-65	32-36	49-53	31-32
	1	11	21	64	38	51	35
Tensile strength, psi	0	2145	2580	1435	1765	1475	2270
	1	2465	2350	1395	1640	1455	1810
Elongation at break	0	505	280	395	250	410	410
	1	560	330	410	300	435	400
Tensile set	0	422	73	17	111	16	429
	1	432	57	14	106	12	208
S-200[1], psi	0	1260	1965	690	1520	760	1330
	1	1205	1550	685	1245	740	1090
Tear strength, ppi	0	390	335	180	300	181	255
	1	496	450	202	305	195	320
Hardness (Duro A-10 sec.)	0	98	76	51	79	54	85
	1	-	64	50.5	64	51.5	65.5
Puncture resistance[2] force, pounds	0	13.9	25.8	44.8	32.9	39.4	47.0
	1	14.8	30.1	49.5	57.0	40.1	49.8
Elongation, in.	0	0.76	0.69	1.22	0.60	1.44	1.04
	1	0.80	0.70	1.20	0.88	1.18	0.98
Volatiles at 105°C	1	0.02	3.55	2.02	12.76	5.54	6.84
Seam strength peel, ppi	0	15.6	40	3.8	30.0	2.5	10.0
	1	10.3[3]	5.1	2.9	3.4	2.0	5.1
Shear, ppi	0	20.2	37.2	30.0	50	14.6	57
	1	11.4	25.6	42.0	40.2	24.3	35

Source: Reference F-6.

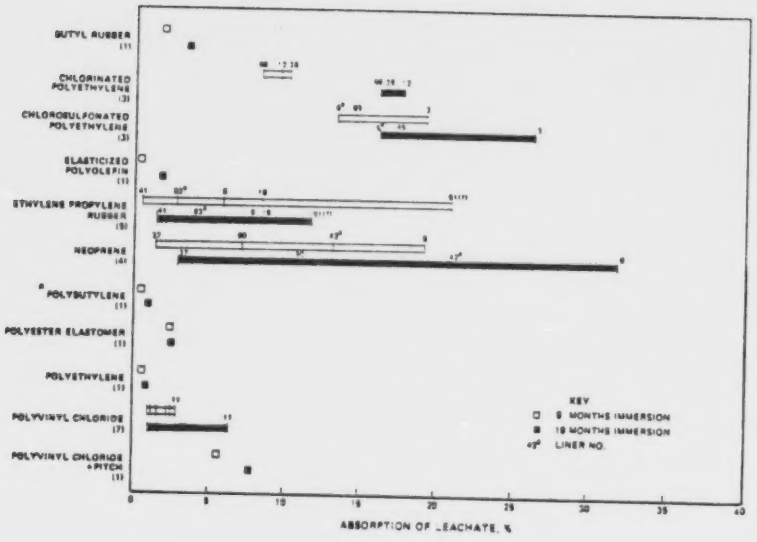
[1]Stress at 200% elongation.
[2]Rate of penetration of probe 20 inches per minute.
[3]Seam in the polyethylene liner used in the steel column. Tabs in the liner specimens mounted in base were too short.

TABLE F-2. SUMMARY OF THE EFFECTS OF IMMERSION OF POLYMERIC MEMBRANE LINERS IN REFUSE LEACHATE FOR EIGHT MONTHS (Ref. F-7)

	No. of Liners in Test	Absorption of Leachate, (%)	%Original value for exposed membrane			Change in Hardness, Points
			Tensile Strength (psi)	Elongation (%)	S-200[1]	
Butyl Rubber	1	1-2	90-97	104-106	85-86	0
Chlorinated polyethylene (CPE)	3	8-10	80-115	64-135	84-123	-5 to -1
Chlorosulfonated polyethylene (Hypalon)	3	13-19	82-124	97-107	47-113	-20 to -4
Elasticized polyolefin	1	0.1	86-94	91-92	102-106	0
Ethylene propylene diene monomer rubber (EPDM)	5	1-13.5	90-91	76-138	98-220	-1 to +2
Neoprene	4	1-19	69-100[2]	82-103[2]	79-102[2]	-11 to +5
Polybutylene	1	0.1	96-99	96-97	99-103	-3
Polyester elastomer	1	2.0	99-115	101-108	95-110	-4
Polyethylene	1	0.6	110-180	96-181	100-116	-7
Polyvinyl chloride (PVC)	7	1-3	91-110	98-129	76-102	-2 to +1
Polyvinyl chloride + pitch	1	6.0	92	109-133	93-108[3]	-2

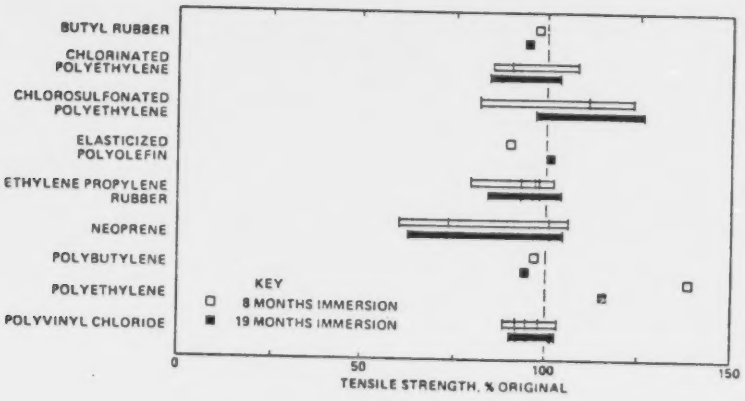
Source: Reference F-7.

[1]Stress at 200% elongation.
[2]Data on fabric-reinforced neoprene liner Number 42 were not included.
[3]S-100 - unexposed specimen broke at 150% elongation.



NOTE: The number of different liners of a given polymer that are included in the test is shown in parentheses (from Reference F-8).

Figure F-1. Swelling of Membrane Liners During Immersion in Leachate for Eight and Nineteen Months.



NOTE: Tensile strength data were obtained by averaging the tests in machine and transverse directions (from Reference F-8).

Figure F-2. Retention of Tensile Strength of Polymeric Membrane Liners on Immersion in Landfill Leachate after Eight and Nineteen Months.

Another EPA study involving the compatibility of liner materials with the leachate from flue gas cleaning sludge leachate (Reference F-9) showed that synthetic membrane liners have reduced tensile strength with increased exposure to the leachate. This FGD sludge leachate may be closer chemically to the FBC waste leachate generated in the current study; however, it should be noted that none of the aforementioned liner studies have been performed with FBC waste leachate. Therefore, the behavior in these studies may not necessarily be consistent with the results of this present study. Controlled conditions (e.g., external pressure, aerobic and anaerobic conditions, immersion technique), also affect the results of these studies.

F.4 Methods

Somewhat different methods are used for the synthetic polymer and bentonite clay parts of the investigation.

F.4.1 Synthetic Polymer Liners

The six organic-based liners were placed in contact with equilibrium leachate from the Exxon PFBC waste. To meet the objective and remain within the program budget it was necessary to develop an accelerated testing procedure. Accelerated interaction implies that measures were taken to increase liner-leachate interaction; however, this study design may not represent the worst case possible, but rather accelerated interaction. Since there are no standard methods for determining liner compatibility, and because the effects of prolonged leachate contact on the liners were unknown, it was difficult to choose the most important design parameters. As a result, the overall plan of the design was to keep the system as simple as possible to avoid problems that might arise over the long term or complicate the interpretation of results.

A batch immersion design, shown in Figure F-3, was used for investigation of the polymer liners. Exxon PFBC waste leachate which had been generated during Step 1 (seven day) of the batch leaching protocol for the program was used for the experiment. The initial leachate composition is given

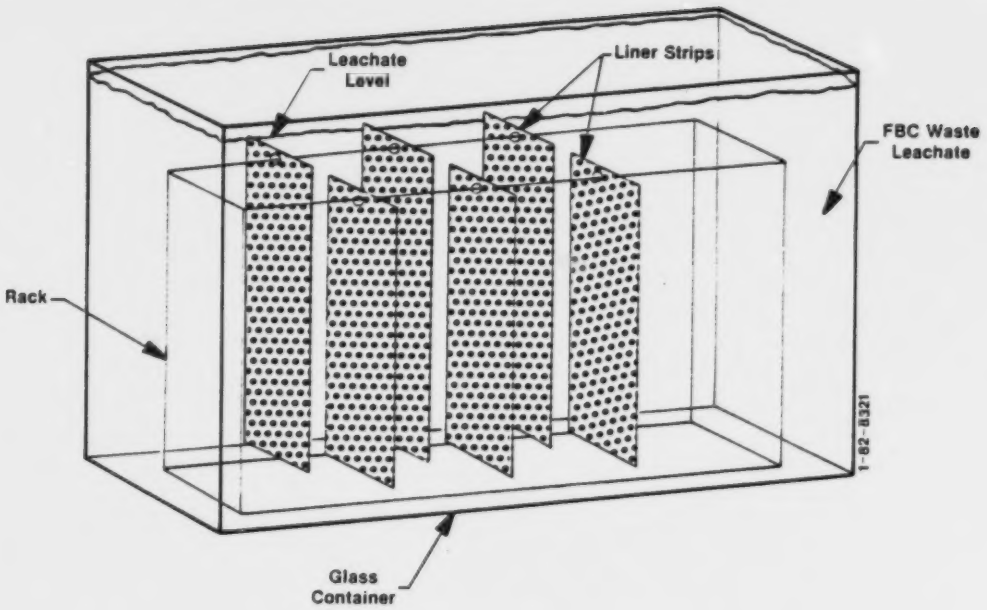


Figure F-3. Batch Immersion Design for Synthetic Liner Exposure to FBC Leachate.

in Table F-3. Both sides of the polymer strips were exposed to the leachate to accelerate degradation. The system was composed of a glass tank fitted with a glass rack to hold the liner strips suspended in the leachate. The tank was filled to the brim and sealed with a glass plate taped to the chamber. Initial air space was minimized, but the air space above the strips increased as samples were removed over the leaching period.

Liner integrity was studied over an eighteen-month period by monitoring the following three parameters:

- Tensile strength of the liner (ASTM Method D-412);
- Total organic carbon (TOC) concentration in the leachate contacting the liner; and
- Scanning electron microscopic observation of the liner surface.

Tensile Strength Measurement

ASTM Method D-412 was used to determine the tensile strength, which was measured for all candidate synthetic liners to monitor the long-term stability of the materials. The tensile strength defines a material's ability to stretch in terms of the stress required to permanently deform it. This stress is equal to or just slightly greater than the strength of the material to maintain its original elasticity. Method D-412 is reported to measure applied force within $\pm$ 2% yielding tensile strength within $\pm$ 10%.

Liner strips of the same length and width (6" x 1") were secured in a tensile strength tester, and the liner strip was stretched until it broke or was permanently deformed. The force necessary to cause breakage or a change in elasticity is the tensile strength of the material.

TABLE F-3. COMPOSITION OF PFBC WASTE STEP 1 USED FOR LINER COMPATIBILITY STUDY

Element/Species	Concentration (mg/L)
Ag	0.003
Al	0.614
As	0.04
B	13.3
Ba	0.171
Ca	1700
Cd	0.0025
Cl	4.46
Cr	0.062
Cu	0.003
F	0.785
Fe	0.224
K	20
Mg	1.91
Mn	0.001
Na	16.7
Ni	0.014
Pb	0.035
pH	11.8
Se	0.06
Si	0.669
SO_4	1230
Sp. Cond.	9530 microsiemens
Sr	293
TDS	3210
TOC	1.1
Ti	0.00001
U	0.15
V	0.005
Zn	0.171

Total Organic Carbon Determination

Single samples of the leachate from each liner vessel were analyzed in duplicate or triplicate monthly for TOC with a Dorhmann Model 52/D TOC analyzer. This instrument utilizes a flame ionization detector to provide linear response up to 10 mg/L carbon concentration.

Scanning Electron Microscopic Investigation

Scanning electron micrographs of representative portions of the polymers were obtained to investigate surface morphology of the strips and the degree of homogeneity over their surfaces.

Small pieces of the polymers were mounted on discs so that representative surfaces were available for examination. Silver paint was used as the adhesive to secure the samples to the base. The mounted samples were placed in a vacuum chamber and coated with aluminum.

The scanning electron microscope (SEM) basically is an extension of the optical microscope, allowing magnifications of the microstructure up to 50,0000 times. For this investigation, an Etec Omniscan scanning elecron microscope was used with a Princeton Gamma Tech Model 1000 energy dispersive X-ray fluorescence spectrometer. Standard conditions used for electron microscope analysis were as follows:

- Voltage, 20kV;
- Working distance, 15 m; and
- Tilt, 30 degrees.

Scanning electron micrographs were produced under two different instrumental conditions yielding micrographs first showing under low magnification of the general surface microstructure, and then under higher magnification to illustrate detailed surface analysis of a specific area.

F.4.2 Bentonite Clay Liner

A sodium-exchanged Wyoming bentonite with high cation exchange capacity and maximum swelling properties was placed in continuous and intimate contact with Exxon PFBC waste leachate. For the clay liner interaction, a 1:10 clay-to-leachate mixture was prepared and placed on a horizontal shaker which agitated the suspension continuously for eighteen months. The following parameters were monitored to assess degradation of the clay:

- Particle morphology (determined by observation by SEM);
- Crystalline structure (measured by X-ray powder diffraction); and
- Aluminum concentrations in the leachate.

Each month, aliquots of both the clay mineral and leachate were taken, and each of the above parameters was determined. SEM and X-ray diffraction allowed direct observation of changes in the clay mineral structure, whereas an increase of aluminum concentration in the leachate gives evidence of the structural collapse of the Al-O network of the clay.

X-Ray Diffraction Analysis

The X-ray diffraction analyses were subcontracted to Baylor University, Waco, Texas. The clay samples were analyzed using a Philips X-ray diffractometer and a special holder designed for use with small samples. This holder allows better interpretation of relative intensities and line shapes than would have been possible with powder camera diffraction.

The X-ray generator and diffractometer were used with a theta-compensating divergence slit assembly. A curved graphic crystal monochromator for a proportional counter was used with a scan speed of 1/2 degree per minute. The samples were ground in an agate mortar and pestle and then brought to equilibrium in a 51% relative humidity desiccator overnight. The samples

were loaded into aluminum holders (cavity of 10 mm x 17 mm x 1 mm) using the McCreary technique (Reference F-10). Samples were prepared so that no preferred orientation of clay particles was visible. The diffractometer chamber was flushed with air at 51% humidity, and the samples were scanned to produce the X-ray patterns. Each diffraction analysis was performed in duplicate.

Aluminum Analysis

Aluminum concentrations in the leachate were measured by inductively coupled argon plasma emission spectrometry (ICAPES), whose detection limit for aluminum is superior to atomic adsorption methods.

Scanning Electron Microscopy and X-Ray Fluorescence Analysis

The same instrument used for the polymer examinations was used for clay examination, but the clay samples were prepared slightly differently. The powdered samples were secured in the same manner, but a standard gold-palladium layer was used as the coating. Each sample was examined at two magnifications, and X-ray fluorescence scans were produced for elemental analysis of bulk areas indicated. The X-ray fluorescence was performed in the scanning electron microscope at the same working distance and tilt.

F.5 Results of Synthetic Liner Studies

F.5.1 Tensile Strength Results

Although a gradual, small loss in tensile strength apparently occurred for all of the candidate liners, no dramatic breakdown was observed for any of the materials tested. The initial strength losses began after about six months of exposure to FBC leachate. The rate of loss was more gradual after 18 months.

Hypalon and PVC liners had the highest original tensile strengths, whereas neoprene and CPE liners had the lowest. Tensile strength retention ranged from approximately 80% for PVC to 60% for CPE and neoprene over the eighteen months of liner exposure to PFBC leachate.

The results of the eighteen months of testing are shown graphically in Figure F-4 and in Tables F-4 through F-9. These tables also show total organic carbon data, which are discussed below. The values shown are averages of measurements performed in triplicate each month. The raw data for the tensile strength are shown in Attachment 1. Some of the earlier force measurements for Hypalon and EPDM rubber were either occurring slightly off scale or at the upper instrumental detection limit of 100 pounds.

The tensile strength of Hypalon decreased from $\geq$2130 psi to a minimum of 1590 psi. The largest changes occurred during the last three months of leaching. Nevertheless, Hypalon retained approximately 75% of its original tensile strength over the 18 months of exposure to the Exxon PFBC leachate, and it appears quite stable and compatible as a liner material for FBC wastes. The Hypalon also underwent an average of only 6% swelling on a mass basis over this time period, which also indicates very little degradation. The final tensile strength measured after leachate interaction was still higher for Hypalon than any other liner material tested.

Second to Hypalon in overall tensile strength was PVC. This liner actually demonstrated an increase in strength during the first eight months of interaction with PFBC waste leachate, and only during the sixteenth month did this material begin to show permanent signs of gradual loss of original tensile strength. The tensile strength for PVC ranged from 1630 psi originally down to 1300 psi, a retention of 80% of the original strength. Again, the period of greatest loss apparently occurred during the sixteenth month of testing. The PVC liner also demonstreated very little swelling, over 7% on a mass basis.

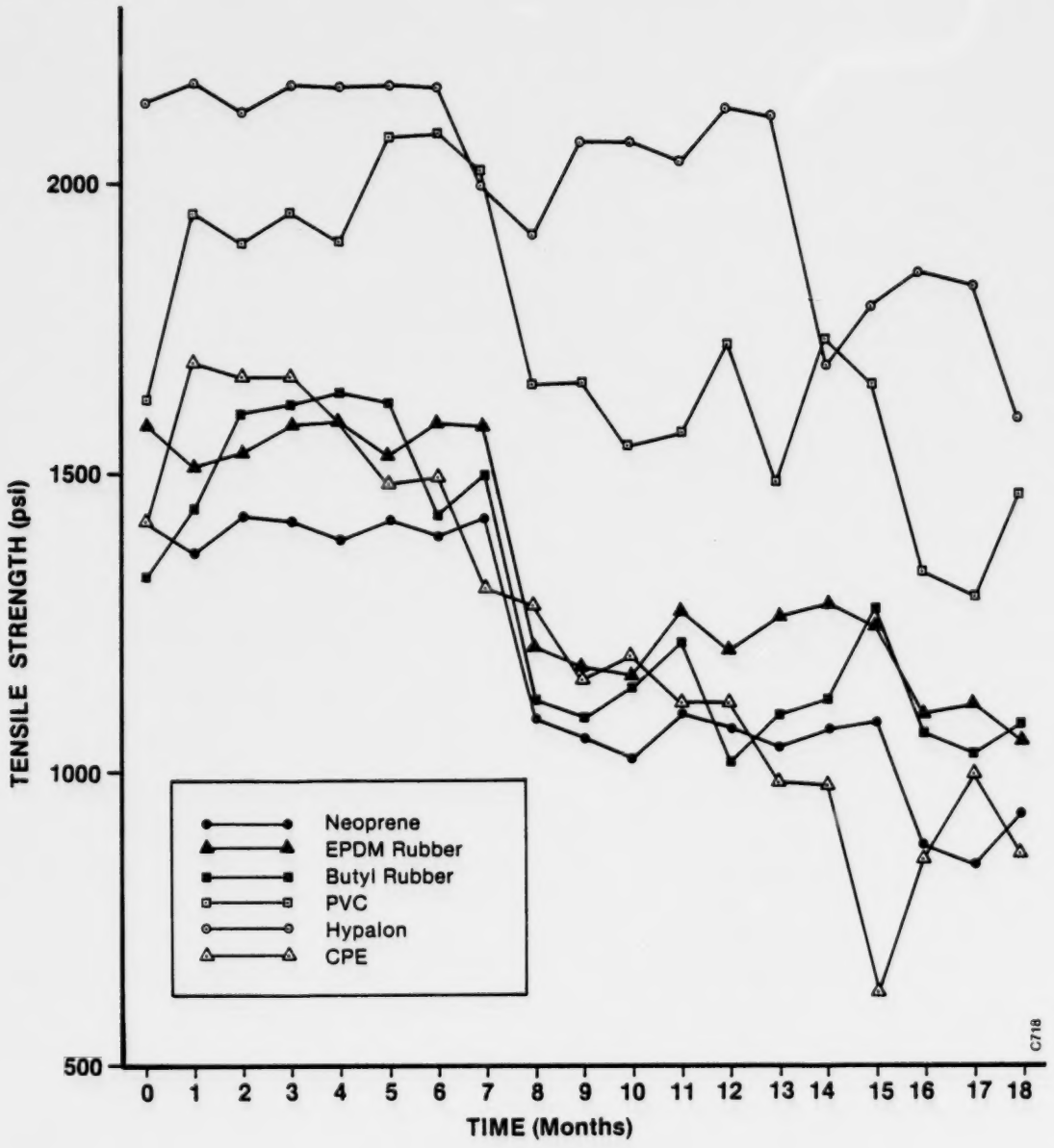


Figure F-4. Tensile Strength of Synthetic Liners as a Function of Exposure Time to Exxon PFBC Waste Leachate.

TABLE F-4. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE NEOPRENE LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	1,420	
1	0.023	0.004	1,370	
2	0.02	0.003	1,430	
3	0.06	0.01	1,420	
4	0.065	0.011	1,390	
5	0.053	0.009	1,430	
6	0.063	0.011	1,400	
7	0.079	0.013	1,430	
8	0.091	0.015	1,090	
9	0.1	0.017	1,060	
10	0.09	0.015	1,020	
11	0.11	0.018	1,100	
12	0.11	0.019	1,070	
13	0.13	0.022	1,040	
14	0.15	0.026	1,070	
15	0.18	0.031	1,080	
16	0.27	0.046	860	4%
17	0.35	0.060	840	
18	0.34	0.059	930	

TABLE F-5. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE POLYVINYL CHLORIDE LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	1,630	
1	1.0	0.096	1,940	
2	1.1	0.11	1,910	
3	1.3	0.13	1,940	
4	1.4	0.14	1,910	
5	1.4	0.14	2,090	
6	1.7	0.16	2,090	
7	1.8	0.17	2,030	
8	1.6	0.16	1,660	
9	–	–	1,660	
10	2.1	0.20	1,560	
11	2.0	0.19	1,560	
12	2.3	0.23	1,720	
13	2.6	0.25	1,470	
14	2.8	0.27	1,720	
15	3.0	0.29	1,660	
16	3.0	0.29	1,340	7%
17	3.6	0.34	1,300	
18	4.0	0.38	1,480	

TABLE F-6. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE CHLORINATED POLYETHYLENE LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	1,430	
1	0.75	0.079	1,570	
2	1.4	0.15	1,670	
3	2.0	0.21	1,670	
4	2.6	0.27	1,600	
5	3.1	0.32	1,500	
6	3.2	0.34	1,500	
7	4.1	0.43	1,300	
8	4.0	0.43	1,270	
9	4.7	0.49	1,200	
10	4.2	0.45	1,200	
11	4.8	0.51	1,110	
12	6.8	0.71	1,110	
13	5.7	0.61	970	
14	6.2	0.66	970	
15	6.9	0.73	630	
16	7.9	0.83	870	31%
17	8.9	0.94	990	
18	10.2	1.1	870	

TABLE F-7. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE HYPALON LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	≥2,130	
1	0.71	0.097	≥2,170	
2	1.2	0.16	≥2,110	
3	1.7	0.23	≥2,170	
4	2.2	0.30	≥2,170	
5	2.5	0.34	≥2,170	
6	2.6	0.35	≥2,170	
7	3.0	0.49	≥2,000	
8	3.7	0.49	1,910	
9	4.4	0.58	≥2,070	
10	4.4	0.59	≥2,070	
11	4.8	0.64	2,040	
12	6.0	0.80	≥2,130	
13	6.0	0.81	≥2,130	
14	6.5	0.87	1,670	
15	7.1	0.95	1,790	
16	7.6	1.01	1,850	6%
17	8.9	1.19	1,830	
18	10.0	1.33	1,590	

TABLE F-8. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE BUTYL RUBBER LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	1,350	
1	0.21	0.035	1,300	
2	0.40	0.066	1,490	
3	0.40	0.067	1,610	
4	0.48	0.079	1,630	
5	0.56	0.093	1,630	
6	0.60	0.099	1,490	
7	0.86	0.14	1,510	
8	1.1	0.19	1,120	
9	1.2	0.28	1,090	
10	1.7	0.30	1,140	
11	1.8	0.34	1,230	
12	2.1	0.36	1,020	
13	2.2	0.36	1,090	
14	2.3	0.39	1,120	
15	2.8	0.47	1,260	
16	2.8	0.46	1,070	3%
17	3.1	0.52	1,020	
18	3.7	0.60	1,080	

TABLE F-9. TENSILE STRENGTH OF LINER AND TOTAL ORGANIC CARBON CONCENTRATION IN LEACHATE AS A FUNCTION OF TIME FOR THE ETHYLENE PROPYLENE DIENE MONOMER RUBBER LINER

Leaching Exposure Time (months)	TOC in Leachate Mass Basis (mg TOC/g liner)	TOC in Leachate Area Basis (mg TOC/in² liner)	Liner Tensile Strength (psi)	Mass Basis (% Swell)
0	–	–	≥1,540	
1	0.13	0.031	≥1,540	
2	0.22	0.054	≥1,520	
3	0.18	0.044	≥1,540	
4	0.19	0.046	≥1,540	
5	0.25	0.06	1,540	
6	0.28	0.067	1,520	
7	0.32	0.076	1,520	
8	0.39	0.095	1,260	
9	0.40	0.095	1,170	
10	0.32	0.078	1,170	
11	0.41	0.097	1,270	
12	0.48	0.12	1,200	
13	0.54	0.13	1,260	
14	0.52	0.13	1,270	
15	0.60	0.14	1,250	
16	0.61	0.15	1,090	16%
17	0.68	0.16	1,100	
18	0.64	0.15	1,040	

All of the rubbery polymers EPDM, butyl rubber, and neoprene) demonstrated very similar tensile strength behavior. The EPDM rubber retained 68% of its original strength (1540 psi to 1040 psi), the butyl rubber retained 80% (1350 psi to 1020 psi), and the neoprene retained 60% (1420 psi to 840 psi). The mass basis swelling for these liners were 16% for EPDM rubber, 3% for butyl rubber, and 4% for neoprene. Although these materials are inferior to Hypalon and PVC, none of them underwent a rapid breakdown during this testing time.

CPE has tensile strength behavior very similar to the rubbery polymers with 61% tensile strength retention (1420 psi to 830 psi), but it was characterized by a mass basis swelling of 31%. This was the greatest swelling observed for any of the materials testeed.

F.5.2 Results of Total Organic Carbon Analysis

The total organic carbon (TOC) concentration in the leachate was measured monthly in each liner system to determine the extent of release of liner material during exposure to the Exxon FPBC waste leachate. This mass of TOC was then normalized to the surface area and the mass of the leached liner. These results are shown in Figures F-5a and F-5b, and Tables F-4 through F-9.

When normalized to the exposed surface area, the Hypalon material showed the greatest loss of total organic carbon to the leachate, followed CPE, butyl rubber, PVC, EPDM rubber, and neoprene. When normalized to the mass of liner material, two slight changes occur in the order of greatest TOC leached from the liners: (1) CPE slightly increased in TOC leached, over Hypalon, and (2) PVC reversed position with respect to butyl rubber.

Neoprene demonstrated the lowest leached TOC, indicating that it had the least organic breakdown into the leaching solution. Second to neoprene was EPDM rubber, which showed an increase of about five times the original TOC leached after one month on an area basis and mass basis. PVC demonstrated a TOC increase of about five times over the first month after

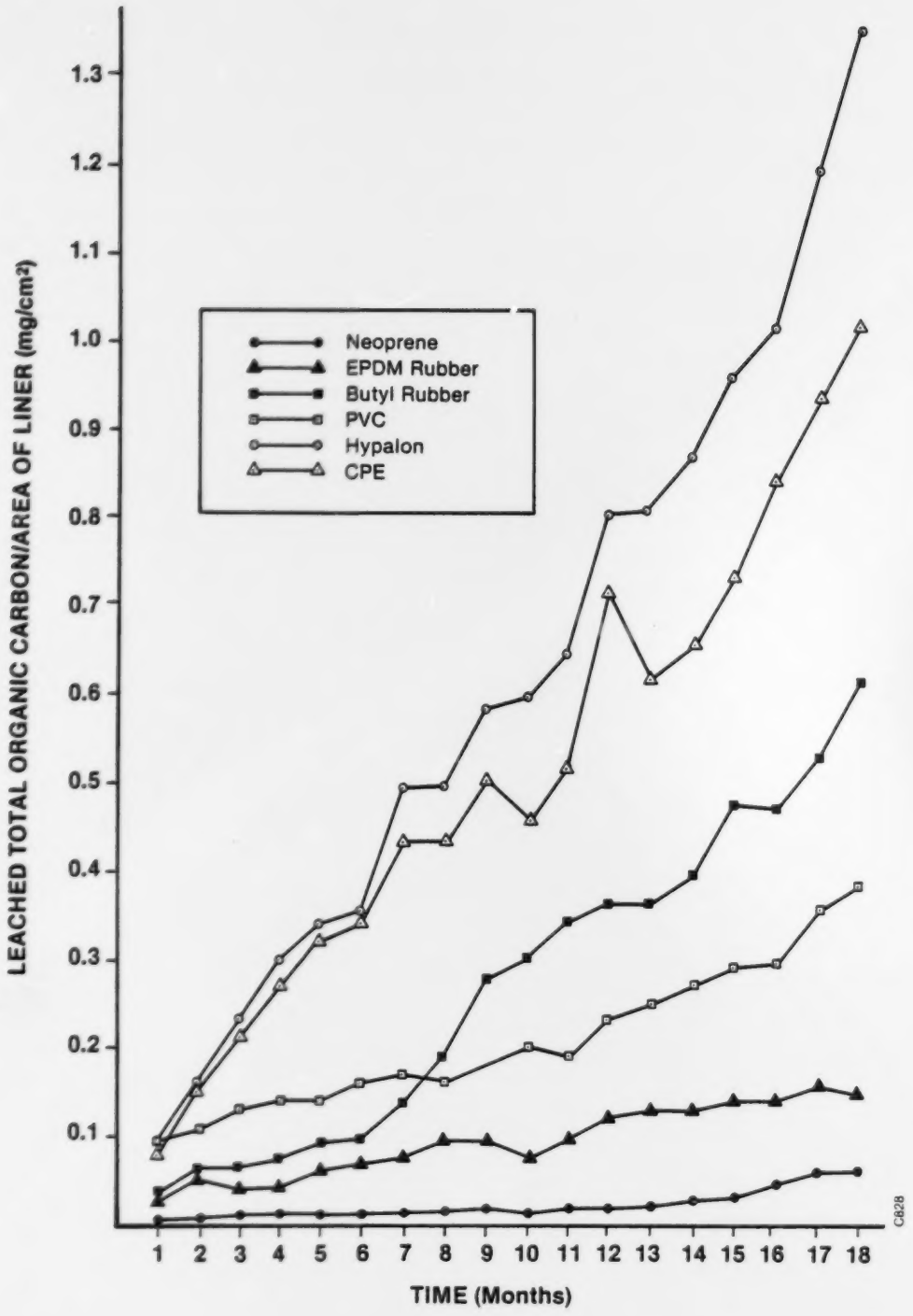


Figure F-5a. Leachable Total Organic Carbon (Area Basis) from Synthetic Liners as a Function of Time.

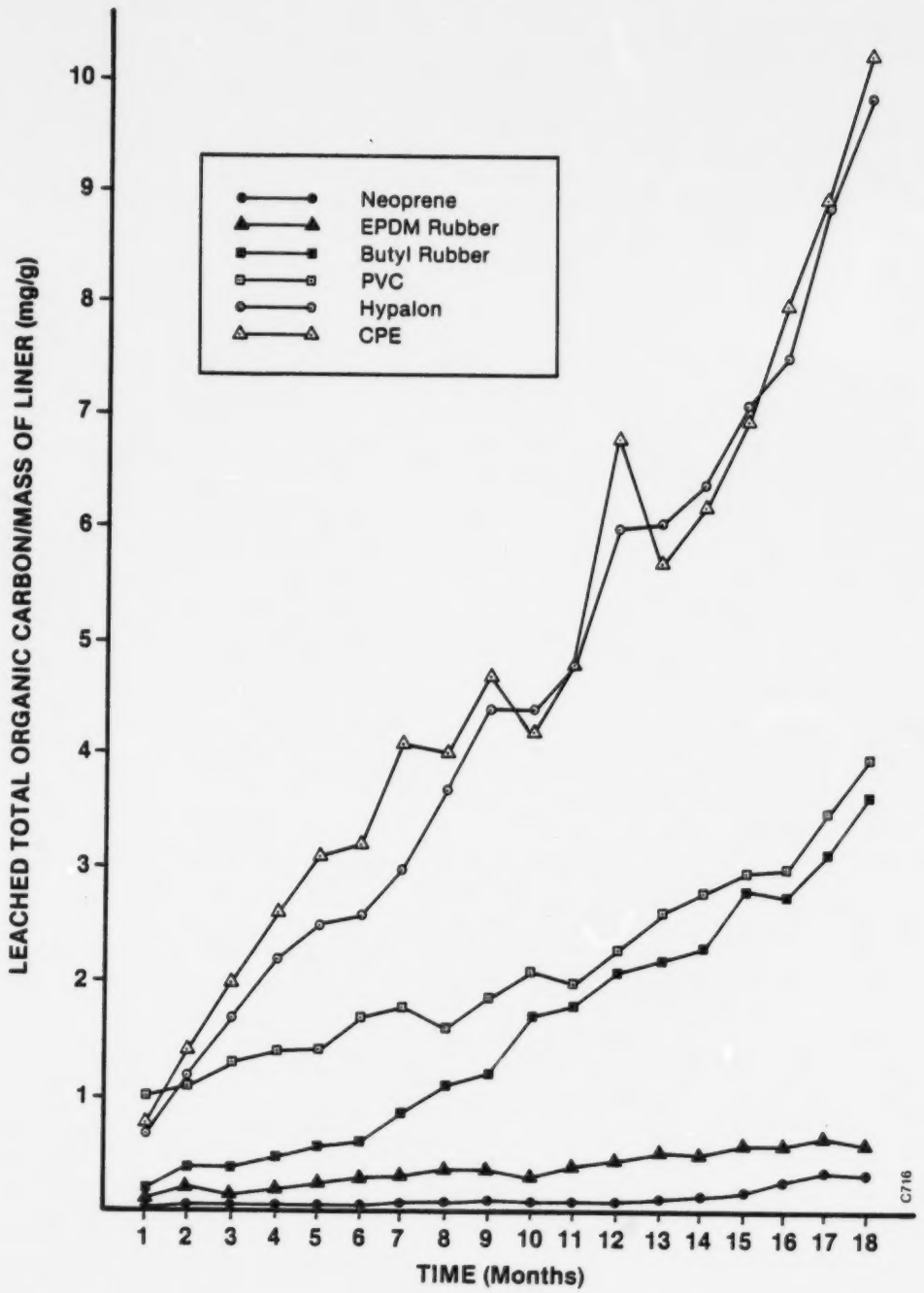


Figure F-5b. Leachable Total Organic Carbon (Mass Basis) from Synthetic Liners as a Function of Time.

18 months of interaction, while butyl rubber showed an increase of 18 times its concentration of TOC after the first month of interaction. The PVC liner and butyl rubber liner were very similar in overall TOC leaching behavior when compared on a mass basis, whereas the PVC liner fell intermediate to EPDM and butyl rubber when compared on a surface area basis. These differences were due to the different densities and thicknesses of these materials.

Hypalon and CPE showed the greatest leachable TOC concentrations. Hypalon increases about 14 times in TOC concentration between the first and eighteenth month on a mass basis and area basis. CPE also increased about 14 times on a mass and area basis during 18 months of interaction with the PFBC waste leachate.

F.5.3 SEM Observations of the Synthetic Liners

In each leachate interaction vessel, one strip of liner was designated for study by scanning electron microscopy (SEM). Two small pieces (1/4-inch buttons) of liner were removed from a specified region of the strip after each month of exposure to the PFBC leachate.

The scanning electron microscope analysis of the polymers was designed to investigate surface degradation and oxidation apparent on the surface of the liner materials. As exposure to the leachate proceeded with time, however, a white precipitate began to crystallize in the bottom of the leaching tanks and to a lesser extent on the surfaces of the liner strips. As the liner strips were allowed to dry before SEM examination, the dry white crystals became more obvious, perhaps as a result of leachate diffusion from the interiors of the liners to the surfaces during drying. Since this material (probably calcium carbonate and possibly calcium oxide) impeded observation of the polymer surfaces, it was necessary to try to remove the crystalline material from the polymer surface without disturbing the fragile, possibly oxidized polymer layers on the surfaces of the liners. Several methods were considered, and the method chosen was to cleanse the polymer surfaces with a gentle stream of deionized water for five minutes. The samples were then

vacuum dried. No physical contact of the surface with any abrasive material, such as brushes or lint-free tissues, was attempted.

This method did remove some of the crystalline material, and in many cases allowed observation of how much of the original surface texture was retained in terms of smoothness and structure. However, the crystals were not sufficiently water-soluble to be completely removed by the treatment method used and still appeared throughout the surfaces of the polymers.

Another problem arose during liner sample coating for SEM observation. Normally a layer of gold-palladium is used to coat the surface. This coating, however, was unsuitable for the polymer materials. As the metal was vacuum evaporated onto the samples, the polymer was in a compressed state in the vacuum chamber. As air was allowed back into the chamber, the elasticity of the polymers allowed expansion of the surfaces back to the original configuration, causing flaking or ripping of the coating. Several other coatings were tried, and aluminum was determined to be the best for these materials.

Each candidate liner material was observed at two magnifications (500X and 5000X) before leaching and again after 16 months of leaching. These two magnifications allowed comparison of general areas at low magnification and fine structure examination at higher magnification. A discussion of results from each liner studied follows below.

Neoprene

The neoprene surfaces were composed of a screen-like grid with high square ridges and lowered square interior areas. Because two different kinds of surfaces were present, both types were investigated. After 16 months of leachate interaction, the neoprene surfaces did not change appreciably. The major difference observed after leaching was the evolution of the calcium-containing crystals on the smooth neoprene surfaces.

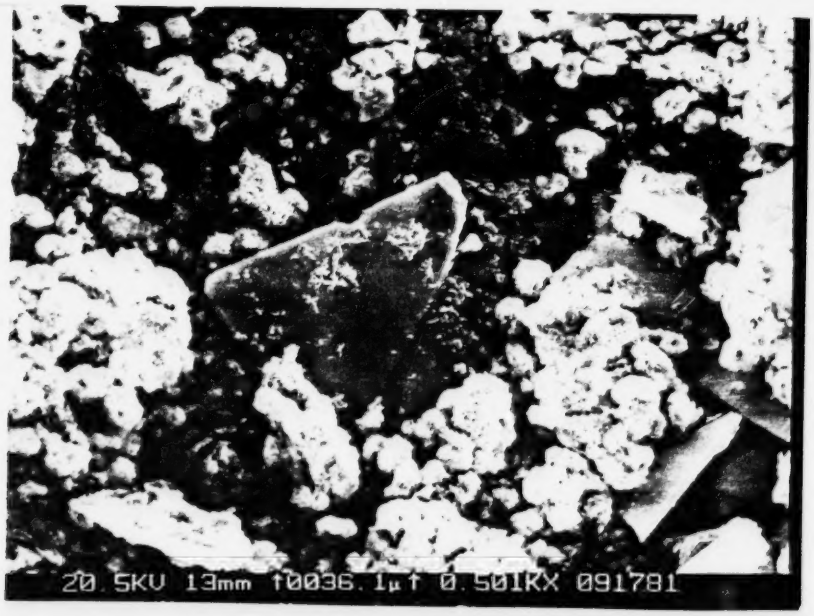
The exterior textured ridges are shown before and after sixteen months of immersion in FBC leachate at low (500X) magnification (Field 1, see Figure F-6). This field shows smooth, very loosely arrayed plates of varying size. Before immersion, the large plates had smooth surfaces, and the predominant orientation resulted from layers of flat surfaces stacked together. After 16 months of immersion in PFBC leachate, the large, flat plates were still present. There was some erosion along the edges of these plates, but the major change was the deposit of crystalline calcium compounds on the plate surfaces.

Field 2 illustrates the lowered interior regions of the neoprene grids at low magnification (Figure F-7). The field shows a more heterogeneous surface before leaching. The plates, although smooth, generally are much smaller and more closely bound. After 16 months of leachate exposure, only the larger plates could be clearly seen. These plates do not show signs of degradation; however, the view is everywhere obstructed by spherical formations of small needles of crystalline calcium compounds.

At higher magnification (5000X), the smooth surfaces of the large plates viewed at low magnification in Field 1 appear quite smooth before exposure to PFBC leachate (Figure F-8). After 16 months of immersion, the surface of the polymer appears to be unaffected by the leachate. A few small crystallites are shown on this Field 1 area which forms the outermost part of the grid.

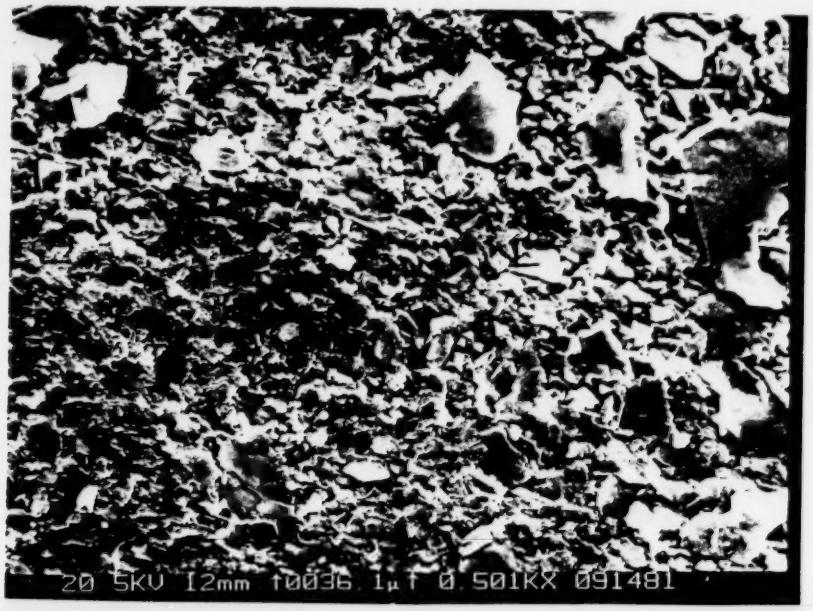
Field 2, the rough interior area of the neoprene grid, is shown at 5000X magnification in Figure F-9. This surface is more heterogeneous than in Field 1. Although the surface is generally flat, the recessed interior grids are replete with long hairline fractures, some crevices, and a few platey areas prior to leachate exposure. After 16 months of immersion in FBC leachate, the polymer (the background) is still smooth; however, crystalline aggregates remaining after rinsing obstruct the view of the polymer surface. The surface itself does not appear to be damaged by PFBC leachate exposure, but is only encrusted with crystalline calcium compounds.

a. Before Immersion

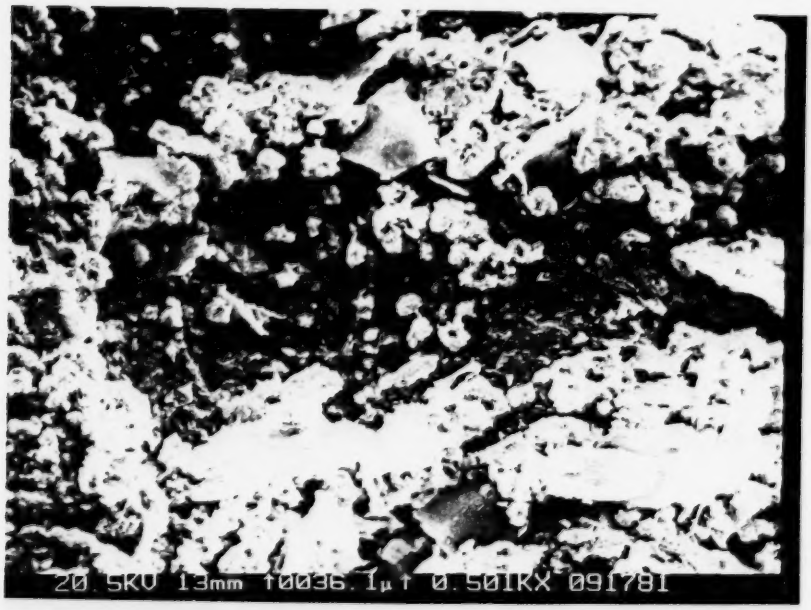


b. After Immersion

Figure F-6. Scanning Electron Micrographs of Neoprene Field 1 (Smooth) Area at Magnification = 500X.

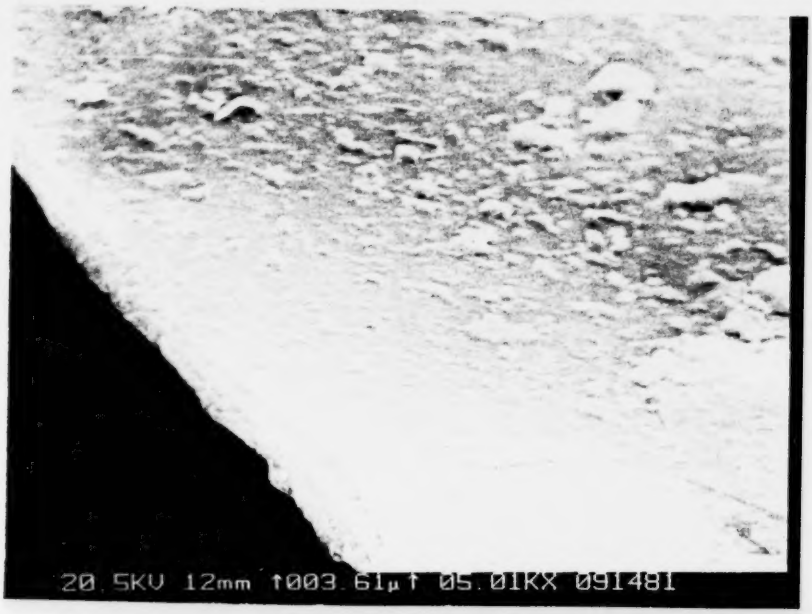


a. Before Immersion

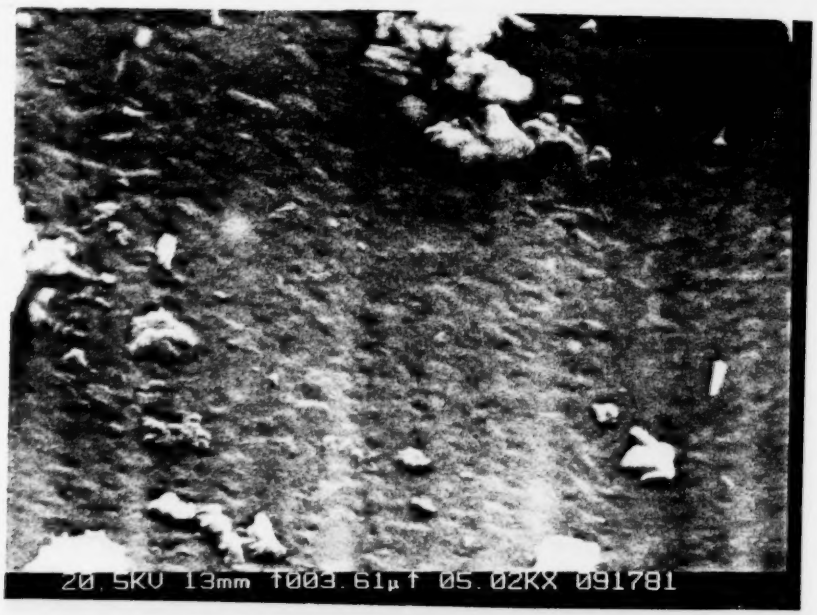


b. After Immersion

Figure F-7. Scanning Electron Micrographs of Neoprene Field 2 (Rough) Area at Magnification = 500X.



a. Before Immersion



b. After Immersion

Figure F-8. Scanning Electron Micrographs of Neoprene Field 1 (Smooth) Area at Magnification = 5000X.

a. Before Immersion

b. After Immersion

Figure F-9. Scanning Electron Micrographs of Neoprene Field 2 (Rough) Area at Magnification = 5000X.

Polyvinyl Chloride

The PVC liner surface was homogeneous over the entire area viewed. Shallow lines in the original polymer appeared to serve as orientation for precipitation of crystalline material during interaction with the PFBC leachate. Although the material looks etched after 16 months of immersion, higher magnification shows complete encrusting of the polymer surface.

In Figure F-10, the PVC surface is shown at low magnification (500X) before and after immersion. At this magnification, both surfaces before and after leaching are similar. The surface of the PVC shows some etching after immersion, but the surface is generally smooth.

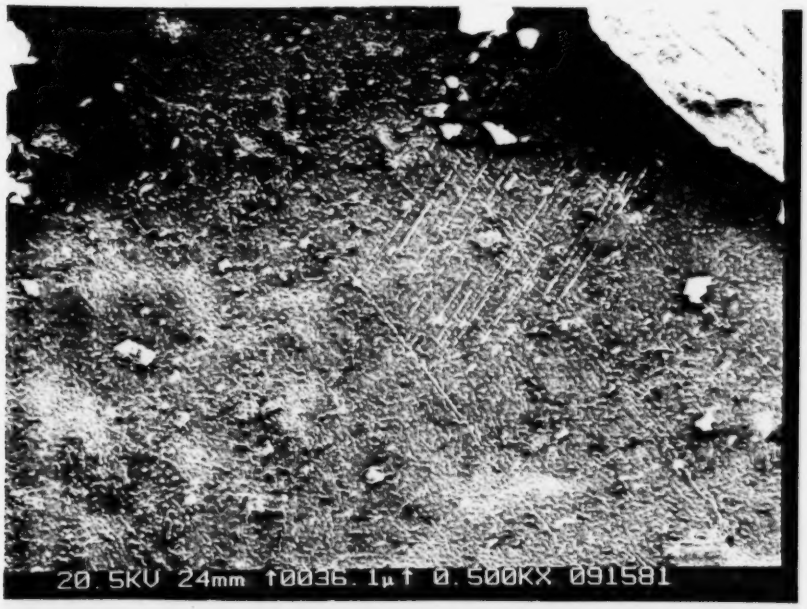
At higher magnification (Figure F-11), it is apparent that the surfaces are quite different before and after immersion. The shallow lines in the original polymer have served to stimulate oriented crystal growth during exposure to the leachate. The surface is totally encrusted, and viewing of the polymer surface is impossible. This calcium compound remained after rinsing, indicating that more surface crystallization from the PFBC leachate is occurring with the PVC than other liner materials.

Chlorinated Polyethylene

The CPE appears to be slightly degraded or oxidized even before exposure to the leachate. It is also the only material to show signs of further degradation with PFBC leachate interaction.

As shown in Figure F-12, at low magnification the original surface of CPE is rough. This material probably has a fiber-reinforced top layer causing the long tubular bumps. After sixteen months of immersion, calcium deposits are prevalent but are cracked, exposing pitted areas in the polymer surface. These areas were not well displayed since the rugged surface caused sample coating problems. As a result, the rugged areas build up a charge in the scanning electron microscope and their photographic representation becomes large white patches with no detail.

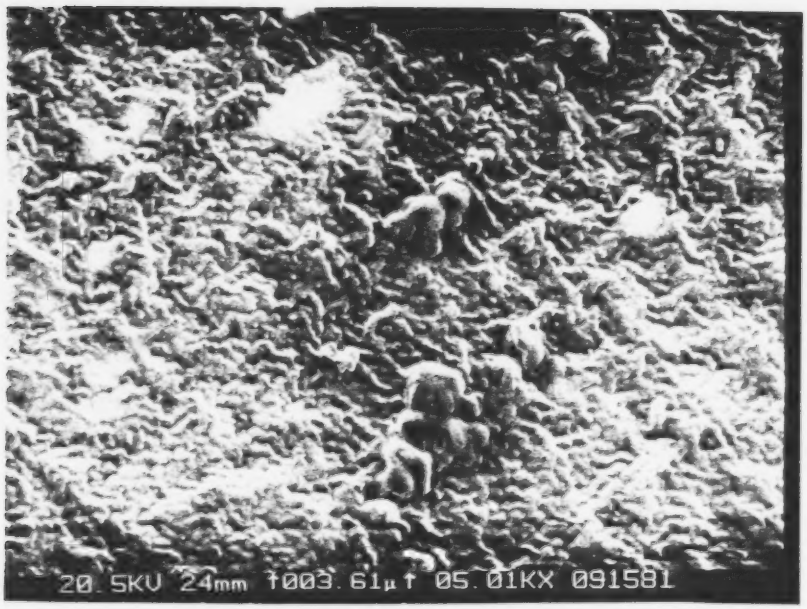
a. Before Immersion



b. After Immersion

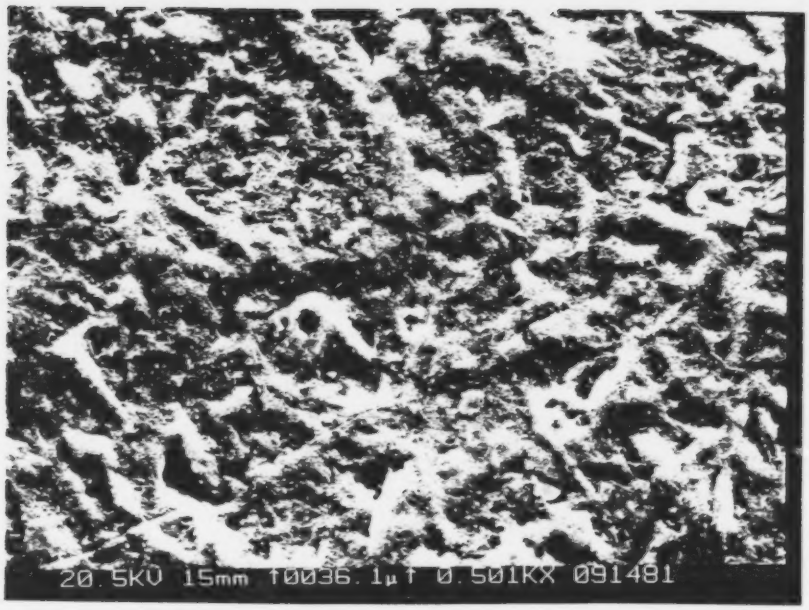
Figure F-10. Scanning Electron Micrographs of Polyvinyl Chloride at Magnification = 500X.

a. Before Immersion

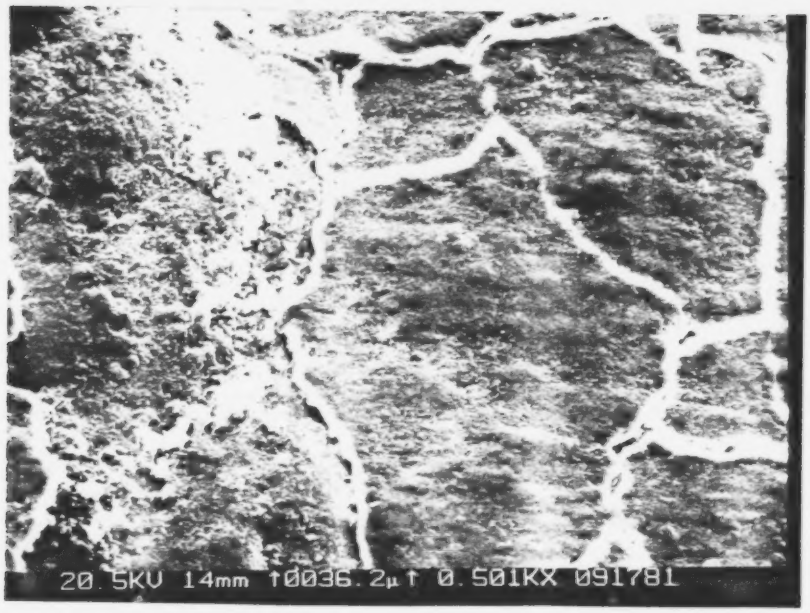


b. After Immersion

Figure F-11. Scanning Electron Micrographs of Polyvinyl Chloride at Magnification = 5000X.



a. Before Immersion



b. After Immersion

Figure F-12. Scanning Electron Micrographs of Chlorinated Polyethylene at Magnification = 500X.

At higher magnification, the gradual degradation of the CPE is demonstrated by the differences in the materials before and after immersion (Figure F-13). Even before interaction, the surface appears to have oxidized areas where degradation has occurred. After 16 months of immersion, the surface is covered with small pits and random crystallization of the calcium compounds. The fibers just below the surface are no longer seen, and the sample has areas where the charge has built up in the scanning electron microscope due to coating problems resulting from the rough surface.

Chlorosulphonated Polyethylene (Hypalon)

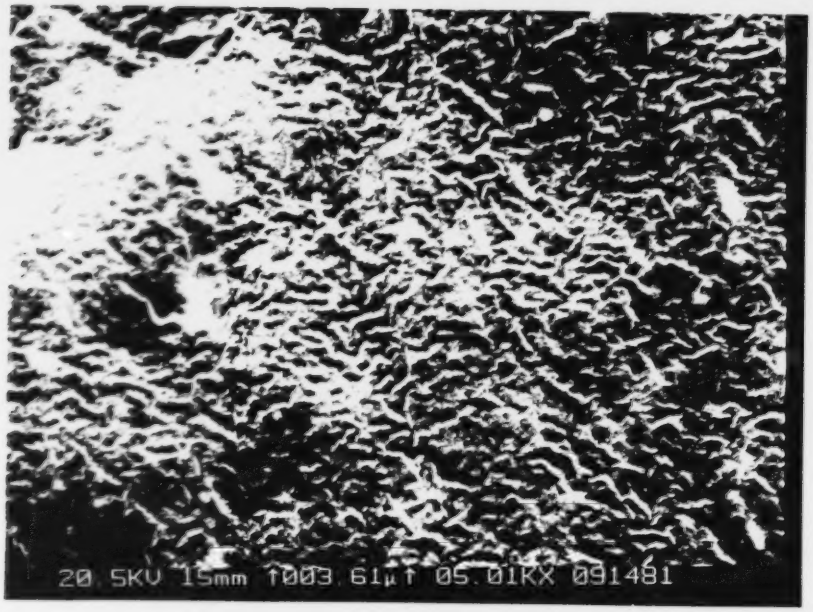
Hypalon also shows some surface degradation before as well as after immersion. Although this material is reinforced with stringy fibers deep within the material, the external appearance is the same over the surfaces above and surrounding the fibers.

Before immersion, the Hypalon appears relatively smooth at low magnification (500X, Figure F-14). The surface does show some indentations and tracks. Tiny particles are present over the surface. After 16 months of immersion, the overall surface became quite rough. The small particles disappeared, and the original craters are deeper.

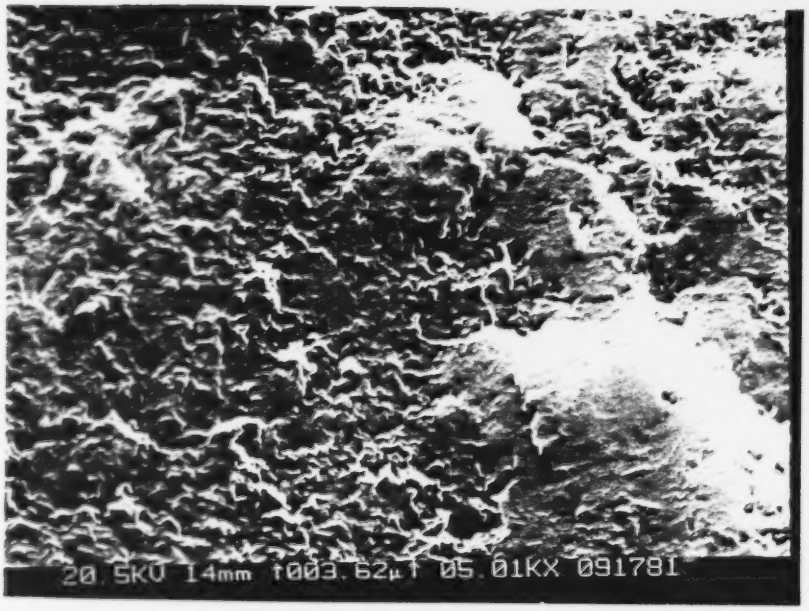
At higher magnification (5000X, Figure F-15), the Hypalon surface appears very rough before immersion. The bright white areas are again due to a charged surface. After 16 months of immersion, the original tracks across the surface have deepened into larger crevices. The surface appears to be gradually "melting" away, with removal of some of the smaller surface particles observed on the original specimen. These micrographs definitely indicate some surface interaction between the PFBC leachate and Hypalon.

Butyl Rubber

Butyl rubber shows similarities to the Field 1 areas of the neoprene, with the difference that the surface of butyl rubber is fiber-reinforced in

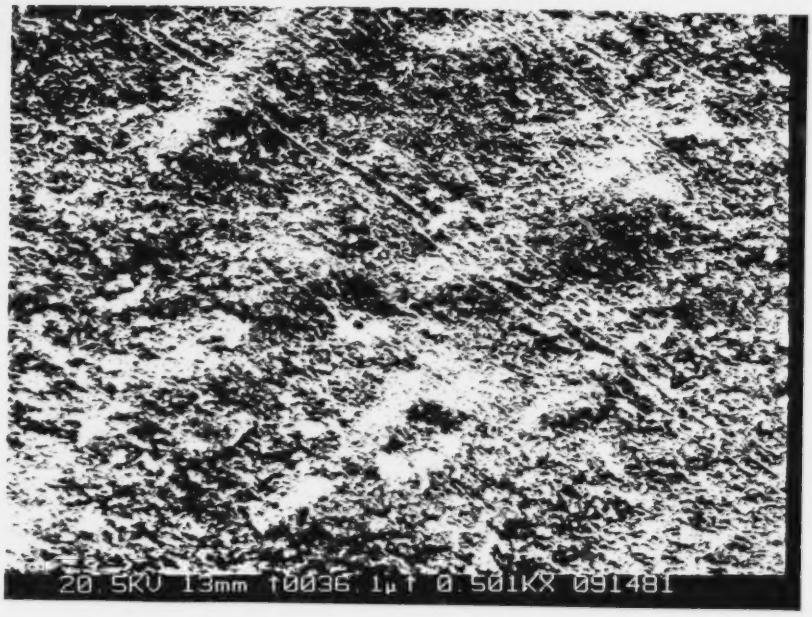


a. Before Immersion

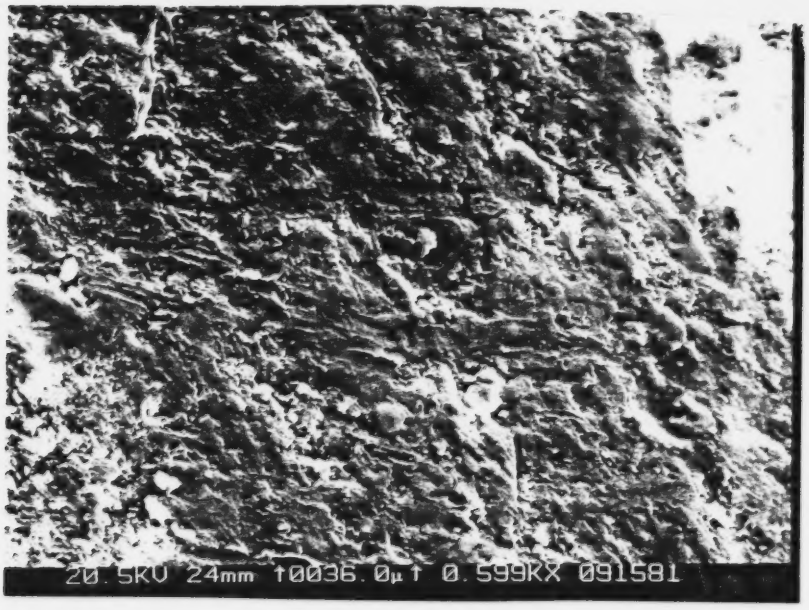


b. After Immersion

Figure F-13. Scanning Electron Micrographs of Chlorinated Polyethylene at Magnification = 5000X.

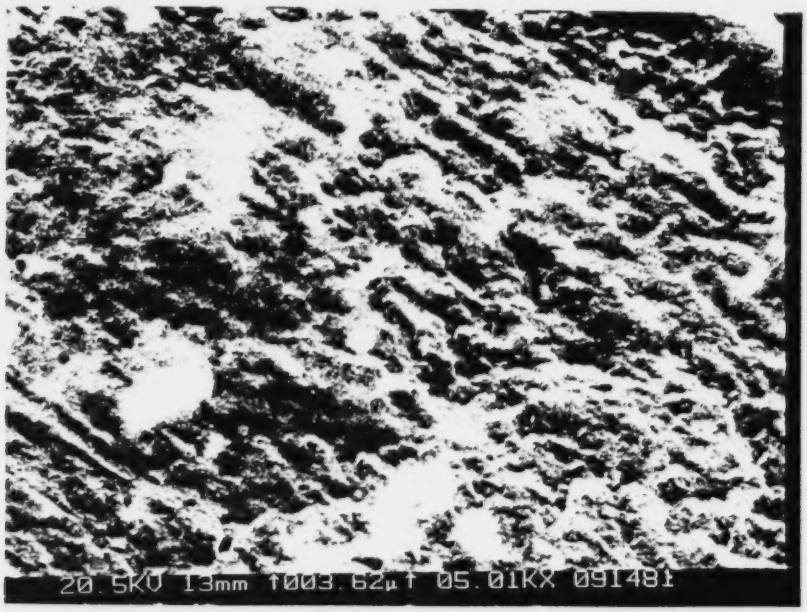


a. Before Immersion

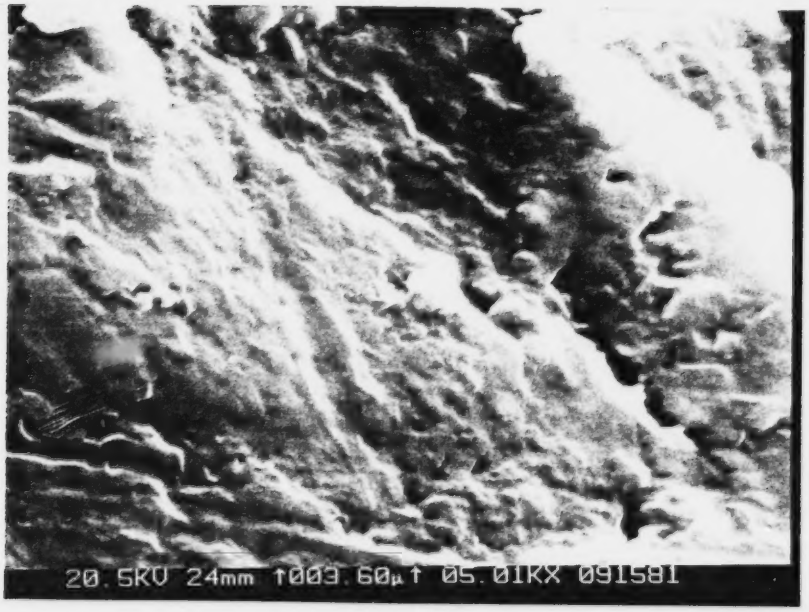


b. After Immersion

Figure F-14. Scanning Electron Micrographs of Hypalon at Magnification = 500X.



a. Before Immersion



b. After Immersion

Figure F-15. Scanning Electron Micrographs of Hypalon at Magnification = 5000X.

this case, and the neoprene surface had a screen-like surface. These rubbery materials appear to attract more precipitation of calcium compounds from the PFBC leachate onto the rubber surface, but they do not appear to degrade or erode.

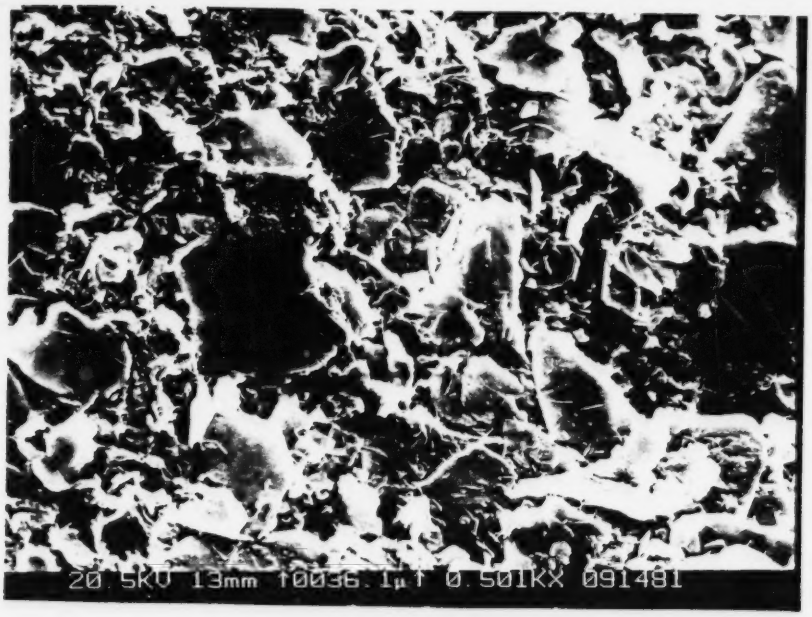
At low magnification (500X, Figure F-16), large flakes of the butyl rubber are oriented in a flat, sandwich fashion on the polymer exterior originally. There is more heterogeneity in platelet size for the butyl rubber than for the neoprene specimen. After 16 months of leachate exposure, discrete platelets are still discernible in the butyl rubber; however, they are completely encrusted with calcium compounds from the leachate.

At higher magnification (5000X, Figure F-17), the original butyl rubber sample shows the smooth platelet surfaces with long fibers actually on the surface. Some of the platelet edges are slightly jagged but the flakes are generally quite smooth. After 16 months of immersion, the outlines of the large particles are completely layered with calcium crystals even after rinsing. A view of the polymer surface is completely obstructed by this material.

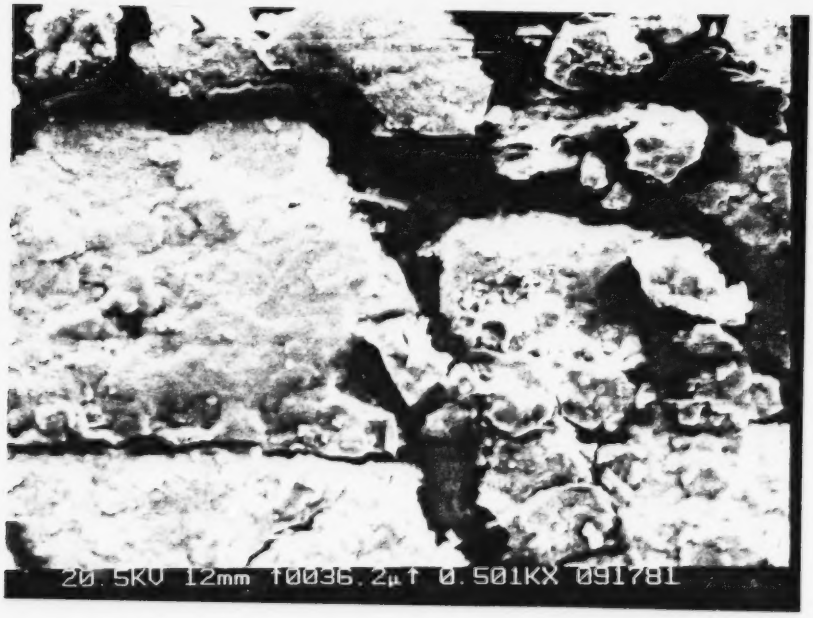
Ethylene Propylene Diene Monomer Rubber

The EPDM rubber is similar to the Field 2 neoprene surface before and after immersion. The EPDM platelets are, however, more closely bound in areas to form a continuous, flat, smooth surface. Encrustation by calcium compounds precludes observations of the polymer surface after sixteen months of immersion.

At low magnification (Figure F-18), the only observable difference between the original material and the immersed EPDM rubber is the profuse buildup of crystals on the immersed sample. The sample before leachate exposure is composed of small delicate lamellae closely held to the surface. After 16 months of immersion, large clusters of needle-shaped crystals cover the entire EPDM exterior.



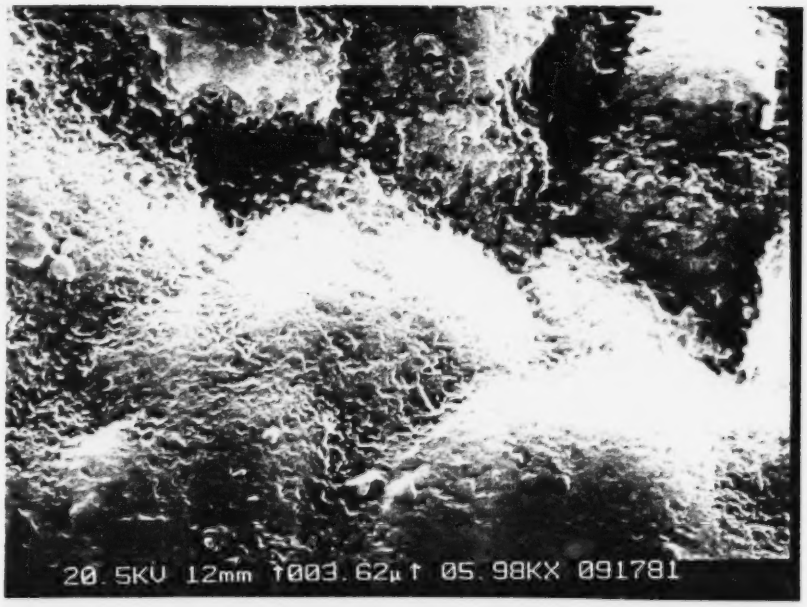
a. Before Immersion



b. After Immersion

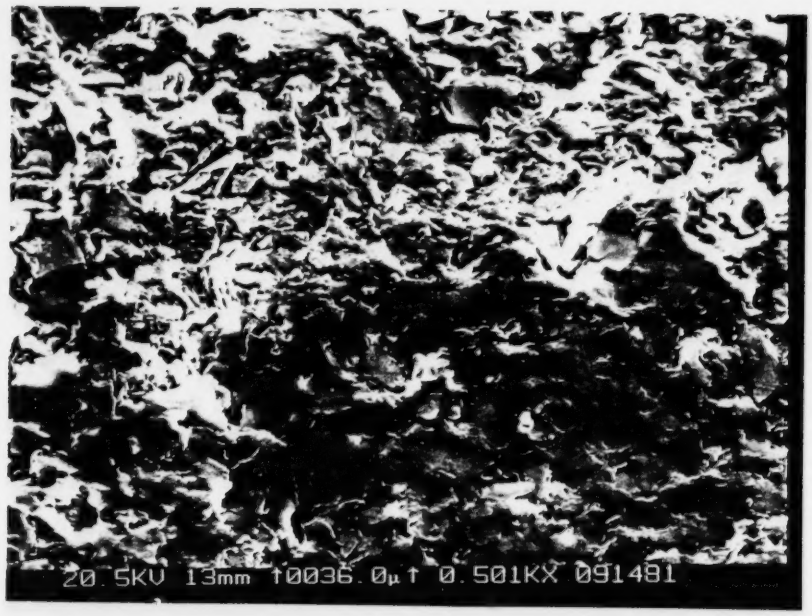
Figure F-16. Scanning Electron Micrographs of Butyl Rubber at Magnification = 500X.

a. Before Immersion



b. After Immersion

Figure F-17. Scanning Electron Micrographs of Butyl Rubber at Magnification = 5000X.



a. Before Immersion

b. After Immersion

Figure F-18. Scanning Electron Micrographs of EPDM Rubber at Magnification = 500X.

At higher magnification (5000X, Figure F-19), the morphology of the original sample is diffuse flakes bound together with deep openings randomly occurring on the surface. After 16 months of exposure, the same basic morphology is still present, but is covered with crystalline spines.

F.6 Results of Clay Liner Studies

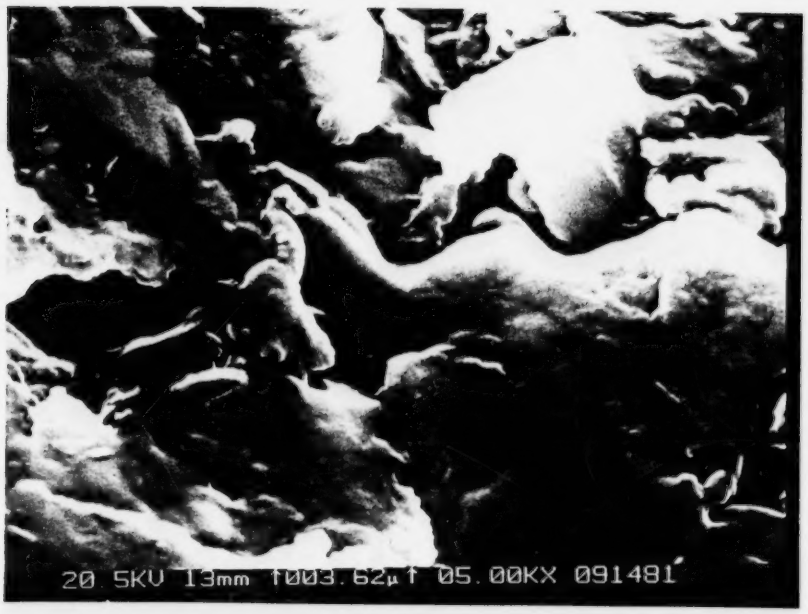
For 18 months a suspension of 1:10 ratio of clay liner material to PFBC waste leachate was shaken continuously. At the end of each month, a uniform aliquot of 50 mL was removed and centrifuged. The liquid from the centrifuged sample was decanted for ICAPES analysis of aluminum concentrations, and the solids were dried in an oven at 40°C. The oven-dried samples were stored in air-tight vials until analyzed by X-ray diffraction and scanning electron microscopy.

F.6.1 Aluminum Concentration Results

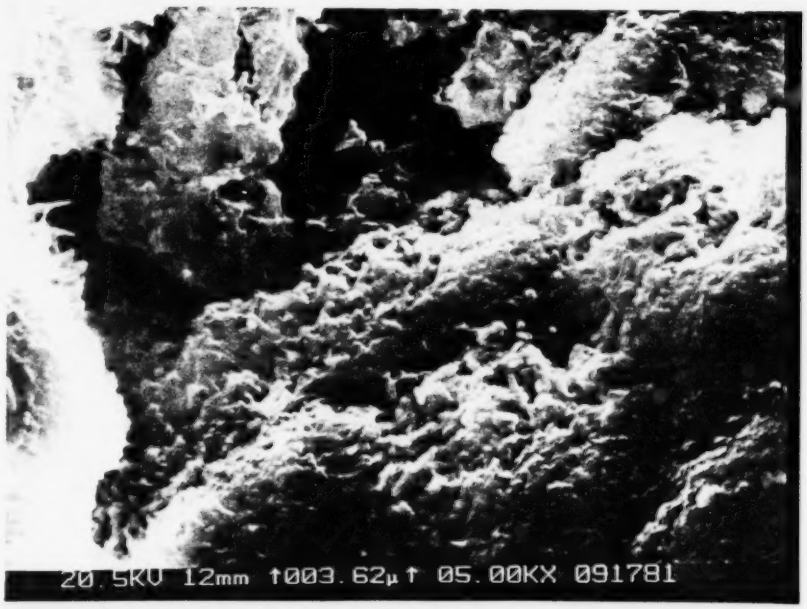
Duplicate analyses of the leachates showed that the concentration of aluminum throughout the experiment was at or below the detection limit and below the level originally present in the PFBC leachate. Thus no chemical breakdown of the internal clay structure was evident over the eighteen months of constant agitation.

F.6.2 X-Ray Diffraction Results

Six of the 18 clay samples collected were examined in duplicate by X-ray diffraction to determine changes in the clay structure due to interaction with the FBC leachate. Relative intensities and line shapes were compared for the various X-ray diffraction patterns as a function of exposure time to leachate. The X-ray diffraction patterns are shown in Figure F-20, and interpretation of these results is given in Tables F-10 and F-11.



a. Before Immersion



b. After Immersion

Figure F-19. Scanning Electron Micrographs of EPDM Rubber at Magnification = 5000X.

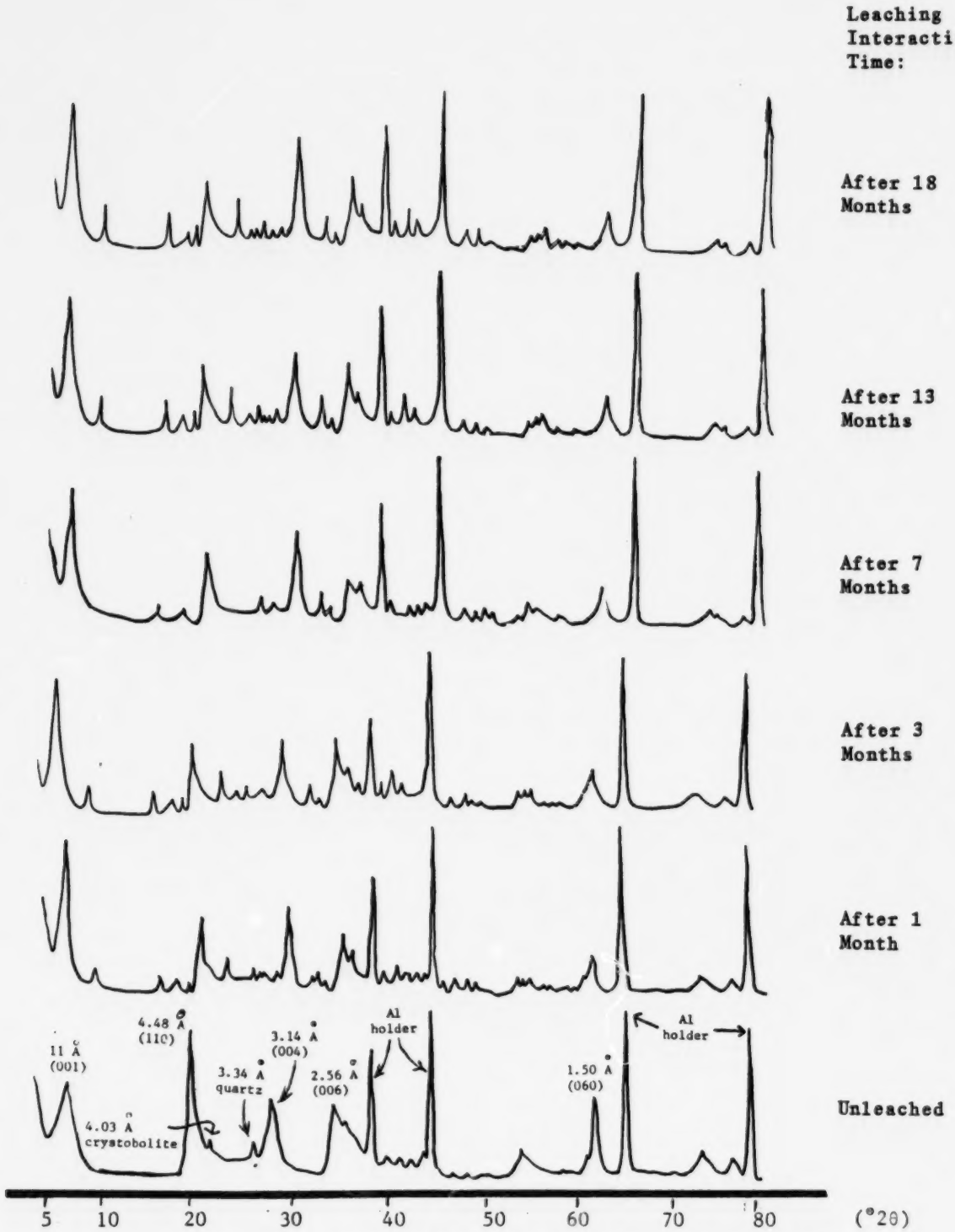


Figure F-20. X-Ray Diffraction Patterns of Clay Liner Samples.

TABLE F-10. D-SPACINGS AND MINERAL IDENTIFICATION FROM X-RAY DIFFRACTION PATTERN FOR THE ORIGINAL CLAY LINER SAMPLE

D-Spacing (A)	Identity
11.0	montmorillonite
4.48	montmorillonite
4.03	quartz
3.34	quartz
3.14	montmorillonite
2.56	montmorillonite
1.500	montmorillonite

TABLE F-11. D-SPACINGS AND MINERAL IDENTIFICATION FROM X-RAY DIFFRACTION LINES AFTER EIGHTEEN MONTHS OF EXPOSURE TO EXXON PFBC LEACHATE

D-Spacing (A)	Identity (Compound-plane)
15.5	montmorillonite (001)
9.60	ettringite
5.60	ettringite
4.92	ettringite
4.69	ettringite
4.48	montmorillonite (110)
4.03	quartz
3.68	montmorillonite (004)
3.66	ettringite
3.60	ettringite
3.46	ettringite
3.34	quartz
3.24	quartz
3.05	montmorillonite (005)
2.77	ettringite
2.69	ettringite
2.61	ettringite
2.56	ettringite + montmorillonite (006)
2.49	ettringite
2.41 (holder peak)	ettringite
2.29	
2.21	ettringite
2.15 (holder peak)	ettringite
1.946	ettringite
1.500	montmorillonite (060)

All of the samples were ground and allowed to come to equilibrium in a 51% relative humidity chamber. Duplicate patterns were obtained for the samples. All patterns were compared to the diffraction pattern obtained for the original sample (prior to exposure to the leachate).

One of the first changes observed is the alteration from a sodium- to a calcium-montmorillonite as reflected by the shift in the 001 line from 11 Å to 15.5 Å. This shift is accompanied by a change in the higher order 001 lines in position, intensity, and half widths.

After one month of interaction with PFBC leachate, a new crystalline phase has formed, and with the exception of the seven-month sample*, there was a gradual increase in the quantity of this phase which is evidenced by the gradual increase in intensities of the various lines associated with the phase. This new phase has been identified as ettringite, a calcium aluminum sulfate silicate carbonate hydroxide hydrate [$Ca_{12}Al_2(SO_4,SiO_3,CO_3)_6(OH)_{24}\cdot 30H_2O$].

Another alteration of the samples exposed to FBC leachate (again with the exception of the seven-month sample) was the gradual change in the shape and structure of the 2.56 Å line from the original sodium-montmorillonite. This change probably indicated a minor degradation in the clay. Another diagnostic area for alteration in clay structures is the 060 line from the original sodium-montmorillonite. However, no appreciable difference was observed in that line; the intensity and structure of the 060 line remained constant. It was characterized by a double peak with a less intense broad peak at around 61° 2θ and a more intense peak at 62° 2θ. This does indicate that the material used for this study was somewhat of a mixed layer-type clay having a mixture of two slightly different structural units.

*This sample may have been dried in an oven at a temperature in excess of 50°C, resulting in structural alteration inconsistent with other data collected both before and after.

The overall indication from the X-ray diffraction analysis was a slow, gradual change of the clay pattern over 18 months of interaction at a 10:1 FBC leachate-to-clay ratio. The leachate/clay interaction resulted in formation of the complex aluminum silicate, ettringite.

F.6.3 SEM and XRF Observations of the Clay Liner Samples

The same samples examined by X-ray diffraction were subjected to scanning electron microscope analysis. Each dried sample was fixed to an SEM sample holder with silver paint, coated with a gold palladium surface, and examined at two magnifications to allow comparison of the overall sample and comparison of single aggregates for loss in fine structure of the montmorillonite. In addition, some areas of the samples were analyzed by X-ray fluorescence in the electron microscope to identify elemental composition of observed particles.

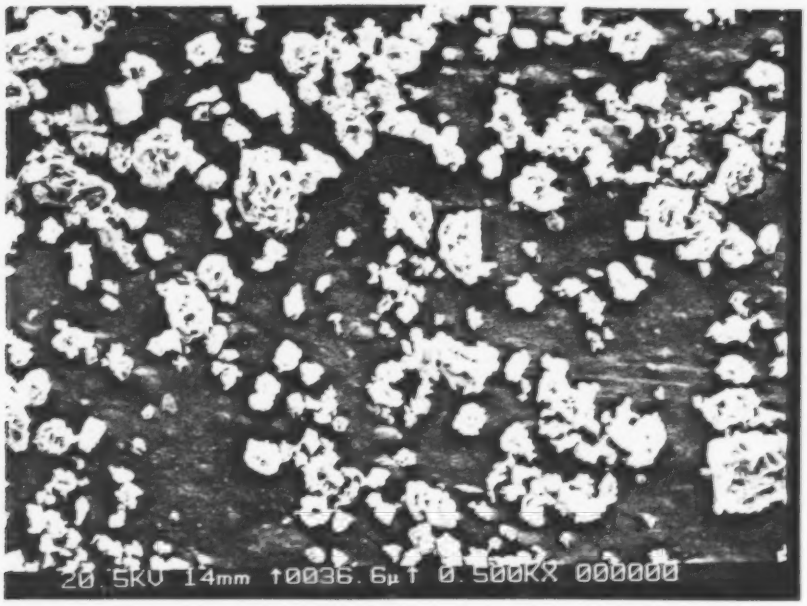
Observations of Original Clay Liner Sample

A photomicrograph of the original sample of centrifuged clay is shown in Figure F-21. The micrograph, at magnification of 5000X, displays the delicate, fluffy rosette morphology common to montmorillonite. This material is uniform with fluffy, thin, filmy aggregates of varying sizes exposed. The edges of the rosette are smooth and continuous.

At lower magnification (500X), also shown in Figure F-21, the distribution of rosettes in the original sample is observed. Prior to leachate interaction, individual rosettes do not aggregate into one cluster; however, some particles present do not have rosette structures.

In Figures F-22 and F-23, the montmorillonite appears to have two morphologies. The morphological difference is also observed as a structural difference in the X-ray data, producing a mixed-layer effect. The familiar rosette is shown in Figure F-22. Adjacent to it is a smooth, irregular clay aggregate. In Figure F-22, a framed area within the rosette was analyzed by X-ray fluorescence (XRF) and the resultant spectrum is presented with the

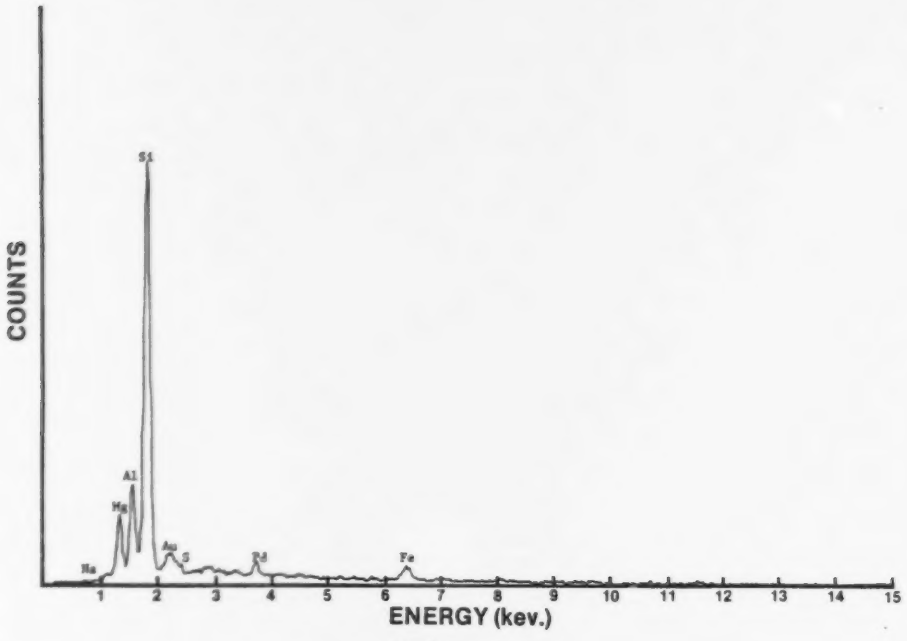
a. Magnification = 5000X



b. Magnification = 500X

Figure F-21. Scanning Electron Micrographs of the Clay Liner Sample Before Leachate Exposure.

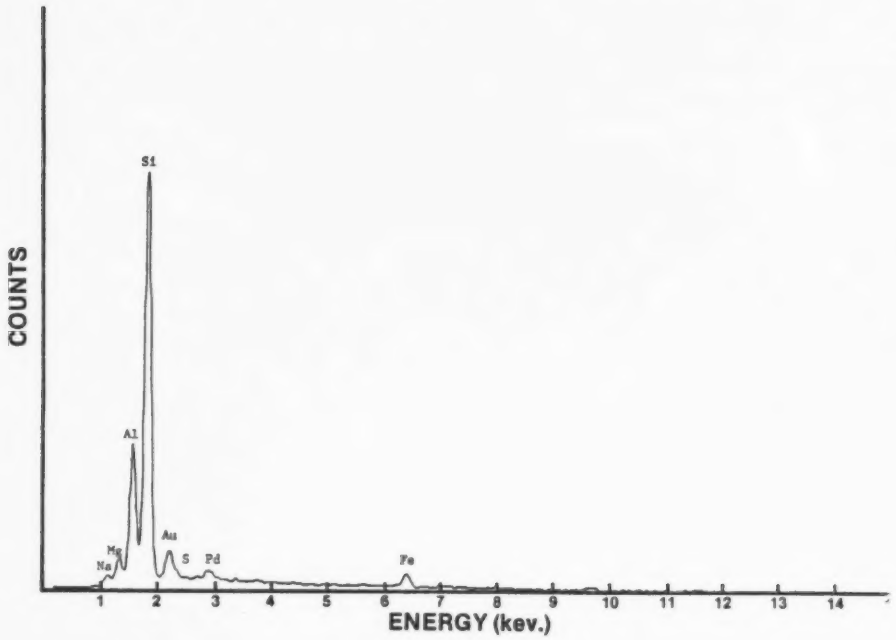
(a.)



(b.)

Figure F-22. (a.) Scanning Electron Micrograph of Original Clay Sample Before Leachate Interation (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum for Bulk Chemical Analysis of Framed Area.

(a.)



(b.)

Figure F-23. (a.) Scanning Electron Micrograph of Original Clay Sample Before Leachate Interaction (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum for Bulk Chemical Analysis of Framed Area.

micrograph. The bulk chemical analysis by XRF revealed the presence of small amounts of sodium, magnesium, and iron in the rosette with greater concentrations of aluminum and silicon. (Gold and palladium in the spectrum are due to the SEM coating and not to the montmorillonite.) Calcium is absent in the bulk analysis of the rosette.

Figure F-23 illustrates the same area shown in the previous micrograph with the emphasis for XRF now placed on the large, smooth particle adjacent to the rosette delineated by the white frame. This particle contained less magnesium and more aluminum according to the XRF analysis. Other elemental concentrations were the same for both morphologies, but two distinct morphologies with different chemical compositions and structures did exist in the unleached clay.

Observations After One Month of Interaction

After the first month of intimate contact with PFBC waste leachate, some expected changes were apparent. At low magnification no major differences are observed. Both morphologies were observed, with the rosette formations dominant. The aggregates shown in Figure F-24 at low magnification may be slightly larger than those illustrated before interaction with PFBC waste leachate, but generally there were no major visible differences from the original material.

Figure F-25 shows the surface of a rosette at higher magnification and the XRF chemistry of the rosette. Visually, the edges appear somewhat ragged. The aggregate retained its general appearance during the first of interaction. However, the XRF scan shows that the bulk chemistry had changed. The montmorillonite became a calcium-exchanged montmorillonite. Practically no sodium is present in the XRF scan, while calcium, which was not originally present, became a major constituent.

When a ratio of aluminum to silicon to calcium was calculated from XRF line intensities, there was a 1:4:2 ratio. This ratio varied from one area to the next, but the increase in calcium was significant.

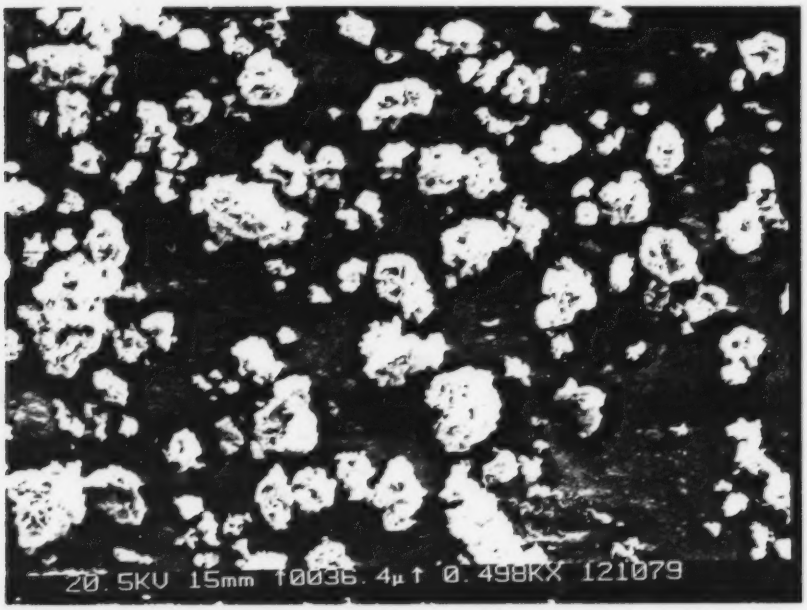
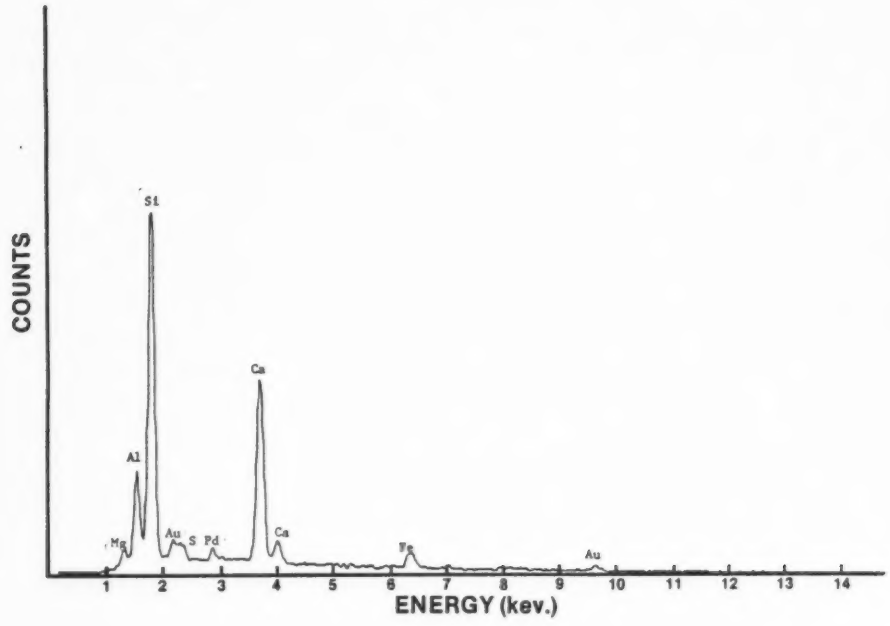


Figure F-24. Scanning Electron Micrograph of Montmorillonite After One Month of Interaction with PFBC Leachate (Magnification = 500X).

(a.)



(b.)

Figure F-25. (a.) Scanning Electron Micrograph of Montomoillonite After One Month of Interaction with PFBC Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Bulk Chemical Analysis of Viewed Rosette.

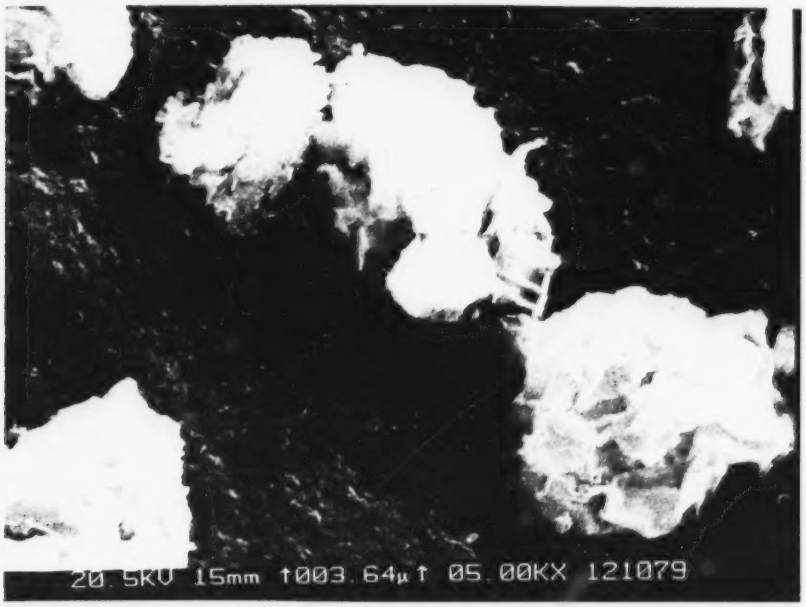
Figure F-26 illustrates the rosette morphology and the smooth-surfaced large particles of another area of the montmorillonite sample. This appearance again illustrates the similarity to the original material. The XRF chemistry, however, was different. The aluminum:silicon:calcium ratio for this surface was 1:4:4. The magnesium concentration was low, and there was no sodium. Iron concentration was higher in the bulk sample shown in this area. There was also some sulfur present due to interaction with the sulfate-rich PFBC leachate.

Figure F-27 shows newly-developed crystallites in the montmorillonite after one month of exposure to PFBC waste leachate. Although the clay itself appeared to maintain structural integrity, new substances were present due to clay-leachate interaction. The XRF analysis of this new crystalline phase showed that the aluminum:silicon:calcium ratio was 1:3:1. There was no sodium present, very little magnesium, and the aluminum concentration was higher than for a typical area of the strip. There was also an increase in the sulfur concentration as shown in the previous XRF scan.

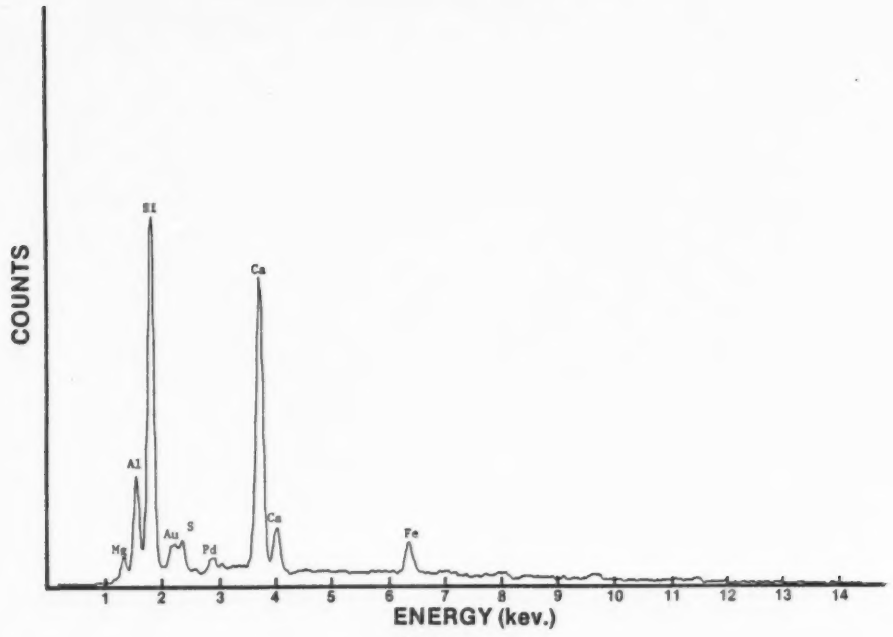
When an XRF scan was performed on the crystallite with more cubic appearance just above the rosette, shown in the framed area in Figure F-28, the chemistry indicated the presence of calcium sulfate or a complex compound containing calcium and sulfur, such as ettringite. There was no sodium, practically no magnesium, and a lower amount of aluminum and silicon present. The concentrations of calcium and sulfur, however, were quite high.

Observations After Three Months of Interaction

After three months of continuous agitation in PFBC leachate, the montmorillonite aggregates showed little change at low magnification (500X). Figure F-29 shows the presence of both original morphologies for the clay, and no degradation is indicated. The aggregates had approximately the same size distribution as before.



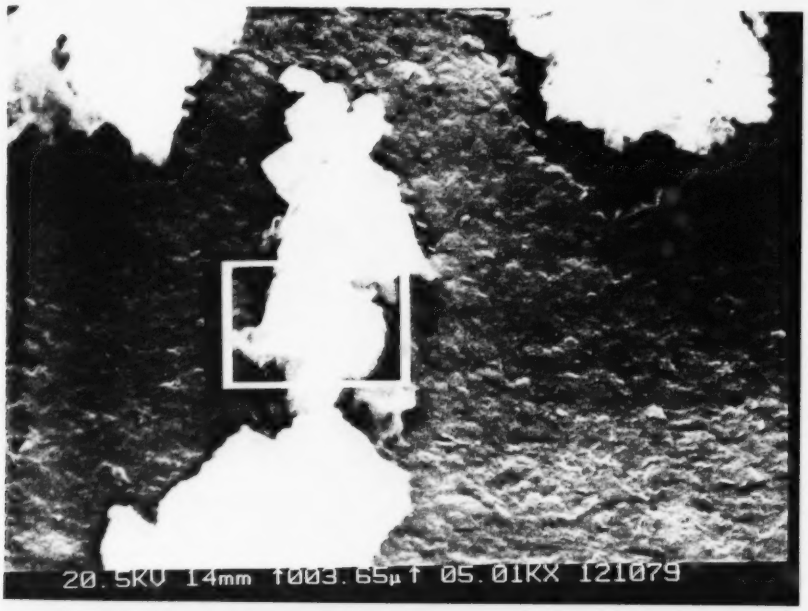
(a.)



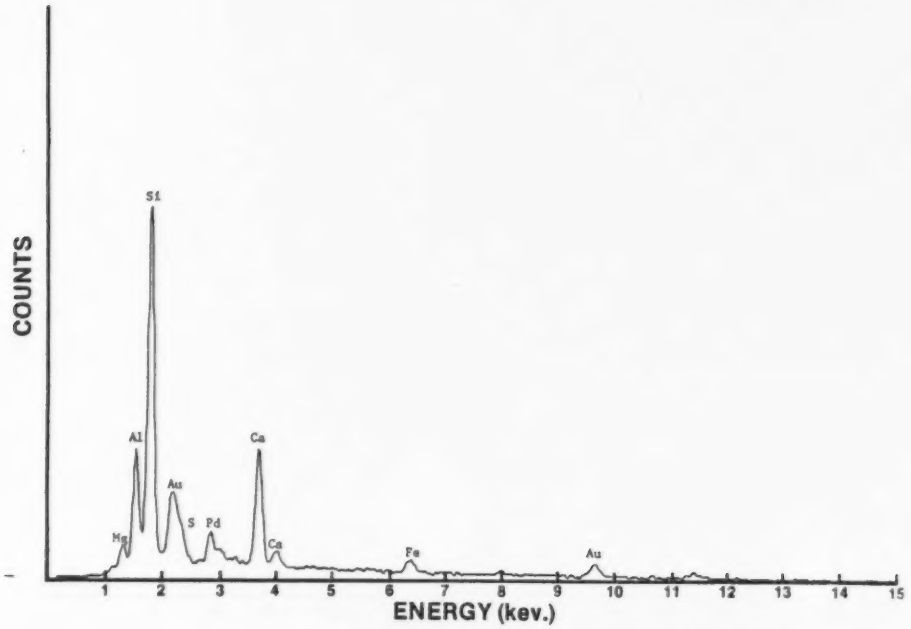
(b.)

Figure F-26. (a.) Scanning Electron Micrograph of Montmorillonite After One Month of Interaction with PFBC Leachate (Magnification = 5000X).

(b.) X-Ray Fluorescence Spectrum of Bulk Chemical Analysis of Viewed Particles.



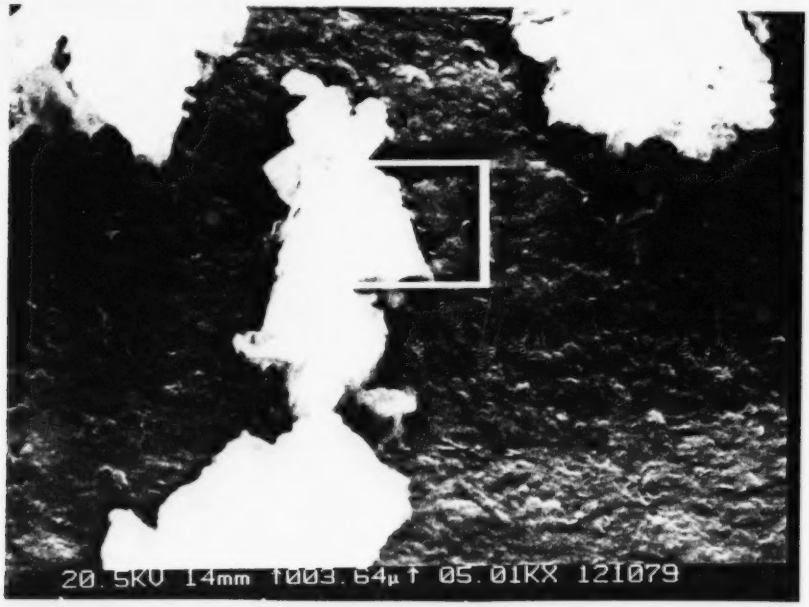
(a.)



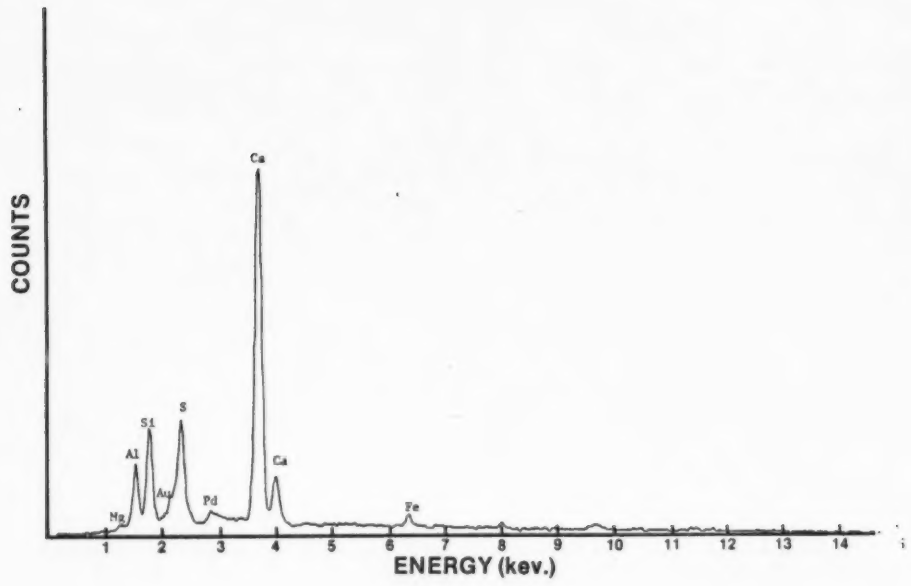
(b.)

Figure F-27. (a.) Scanning Electron Micrograph of Montmorillonite After One Month of Interaction with PFBC Leachate (Magnification = 5000X).

(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Crystallite.



(a.)



(b.)

Figure F-28. (a.) Scanning Electron Micrograph of Montmorillonite After One Month of Interaction with PFBC Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Crystallite.

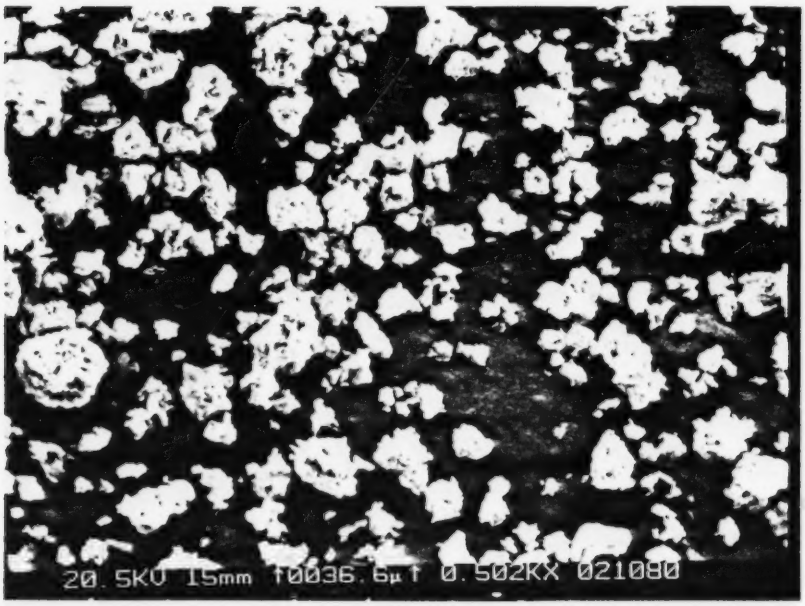


Figure F-29. Scanning Electron Micrograph of Montmorillonite After Three Months of Interaction With PFBC Leachate (Magnification = 500X).

At higher magnification (5000X, Figure F-30), the familiar rosette is as seen throughout the clay sample. The clay surface did not undergo any apparent breakdown. The XRF scan shows the chemistry of an aluminosilicate containing some magnesium and iron. Compared to the original composition, the calcium had replaced the sodium ions in the exchange site, and the sulfur concentration was gradually rising.

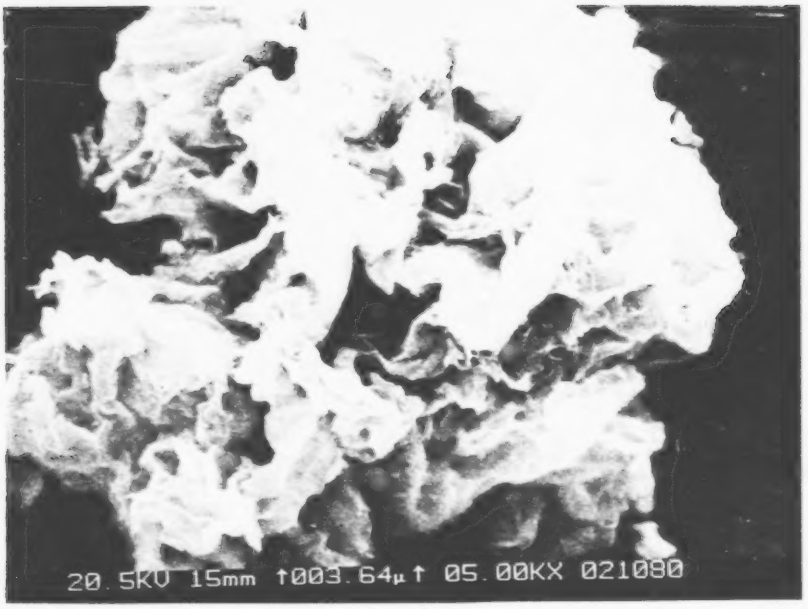
Figure F-31 depicts a new crystallite formed among the clay aggregates. These crystallites were sparsely distributed over the surface and were probably calcium carbonate or calcium oxide. The Al:Si:Ca ratio indicated by the XRF analysis was 1:3:13. Sodium and magnesium were absent, and the aluminum, sulfur, and silicon concentrations were extremely low. The only major constituent detectable by XRF was calcium. This calcium compound was probably a chemical yield from the PFBC waste leachate.

Observations After Seven Months of Interaction

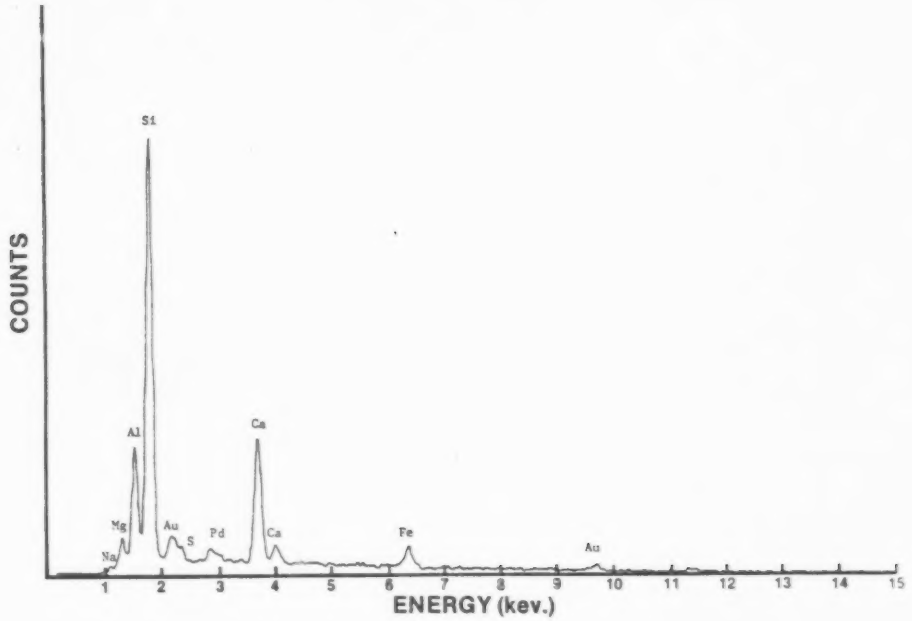
After seven months of continuous shaking in a suspension of PFBC waste leachate, no major changes occurred. The clay appears visibly unchanged. Some of the thin filmy edges of the rosettes were possibly more closely bound.

At low magnification (500X, Figure F-32), the clay appeared normal. The surface was covered with large to small aggregates of smooth particles and fluffy rosettes.

At higher magnification (5000X) the clay had the same appearance. The magnesium concentration reflected in the XRF scan for the particle shown in Figure F-33 had an Al:Si:Ca ratio of 1:9:3. The aluminum concentration was low and the magnesium concentration was a little higher than normal. The calcium sulfate may be going in and out of the aqueous phase and onto the clay surface. This area did not show appreciable sulfur concentrations.

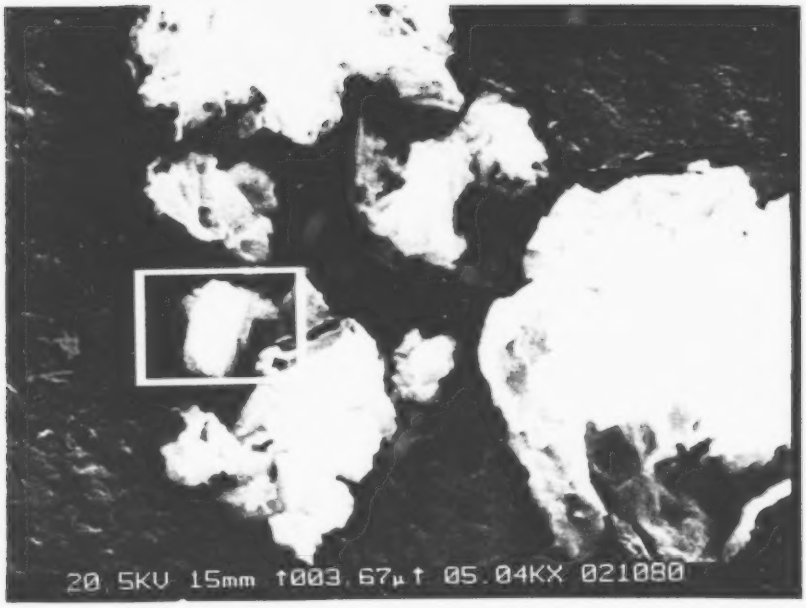


(a.)

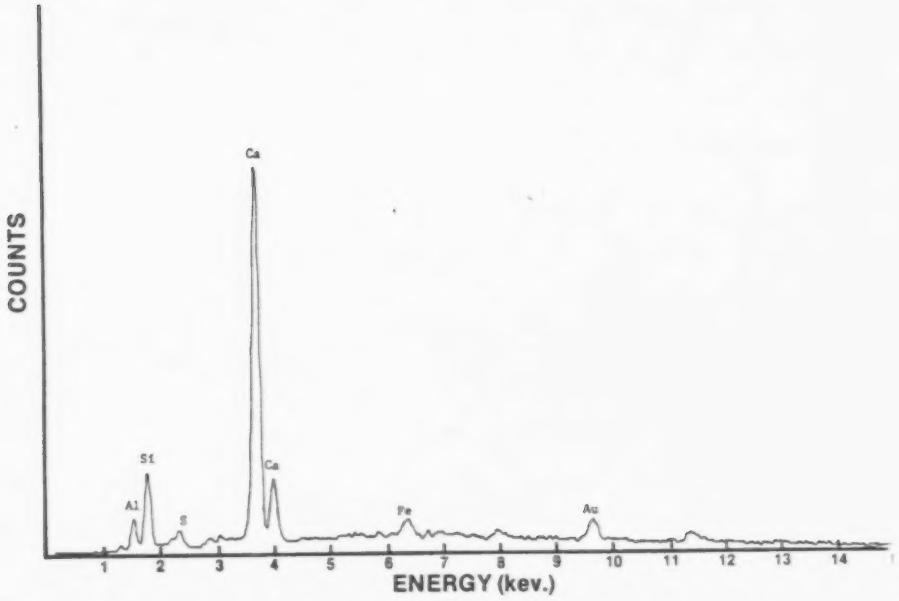


(b.)

Figure F-30. (a.) Scanning Electron Micrograph of Montmorillonite After Three Months of Interaction with PFBC Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Chemical Analysis for the Viewed Rosette.



(a.)



(b.)

Figure F-31. (a.) Scanning Electron Micrograph of Montmorillonite After Three Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Crystallite.

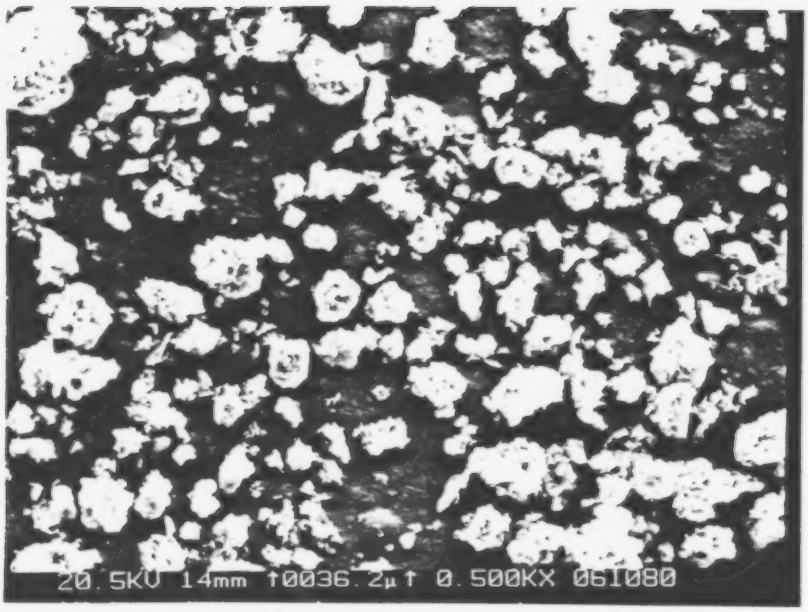
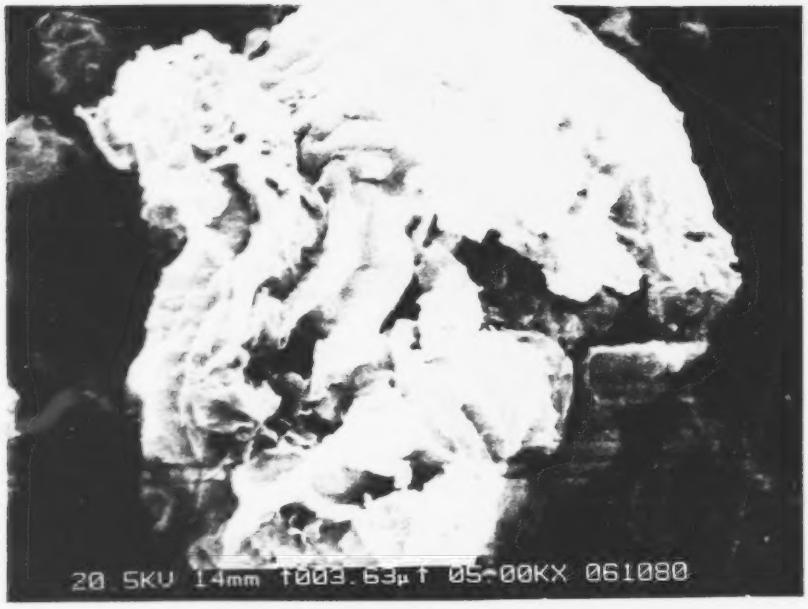
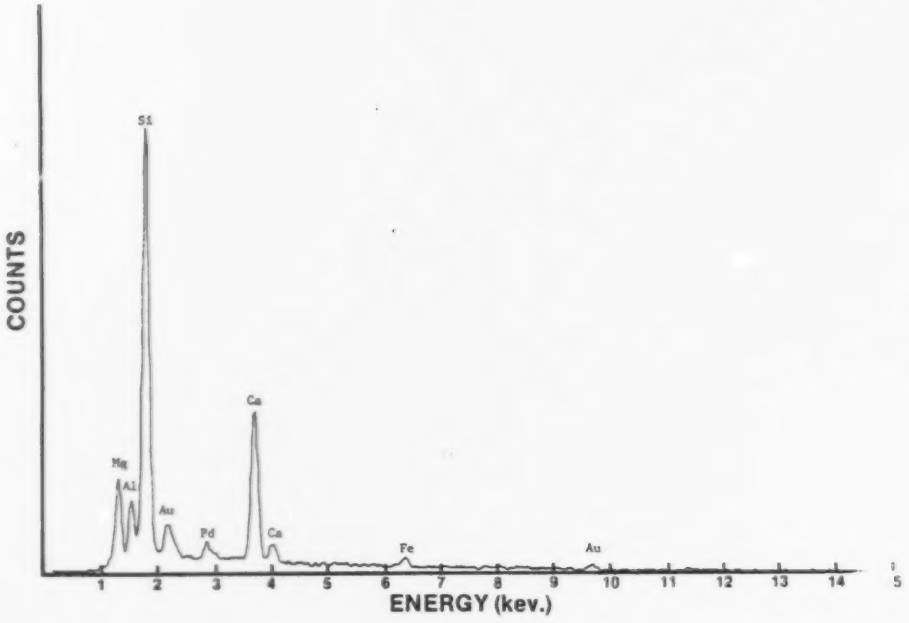


Figure F-32. Scanning Electron Micrograph of Montmorillonite After Seven Months of Interaction with PFBC Waste Leachate (Magnification = 500X).



(a.)



(b.)

Figure F-33. (a.) Scanning Electron Micrograph of Montmorillonite After Seven Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Chemical Analysis for the Viewed Rosette.

Observations After 13 Months of Interaction

After continuous agitation in FBC waste leachate for 13 months, there was still no detectable alteration of the montmorillonite. There was increased presence of a crystalline material tentatively identified as ettringite, and which may have been a reaction product of the clay and the calcium and sulfate compounds from the FBC leachate.

The micrograph of the montmorillonite at low magnification (500X, Figure F-34), looks much the same as the other samples of the montmorillonite. There may be a slight tendency toward larger aggregates.

When viewed at higher magnification (5000X, Figure F-35), the clay remained unchanged. The XRF scan for the area viewed indicated high silicon content, average aluminum content, and moderately low calcium and magnesium in the bulk sample. The clay itself was fairly free of sulfur. Only new crystallites separate from the clay aggregates appeared to have high concentrations of sulfur compounds.

A complex crystallite containing a large amount of sulfur is shown in Figure F-36. This crystallite occurred separate from the clay aggregates. Its appearance was similar to the smooth montmorillonite aggregates, except that the crystallite was somewhat smaller. Its XRF scan showed an Al:Si:Ca ratio of 1:3:1, with a large concentration of sulfur. This material was probably again ettringite. This area also showed a small concentration of iron in the bulk sample.

Another crystalline phase separate from the clay aggregates is shown in Figure F-37. This material was almost cubic in morphology. Its XRF scan showed a high concentration of calcium and sulfur with lower silicon and aluminum concentrations. No sodium or magnesium was indicated in the crystal shown in the white framed area. The ratio of Al:Si:Ca in this crystal was 1:2:4.

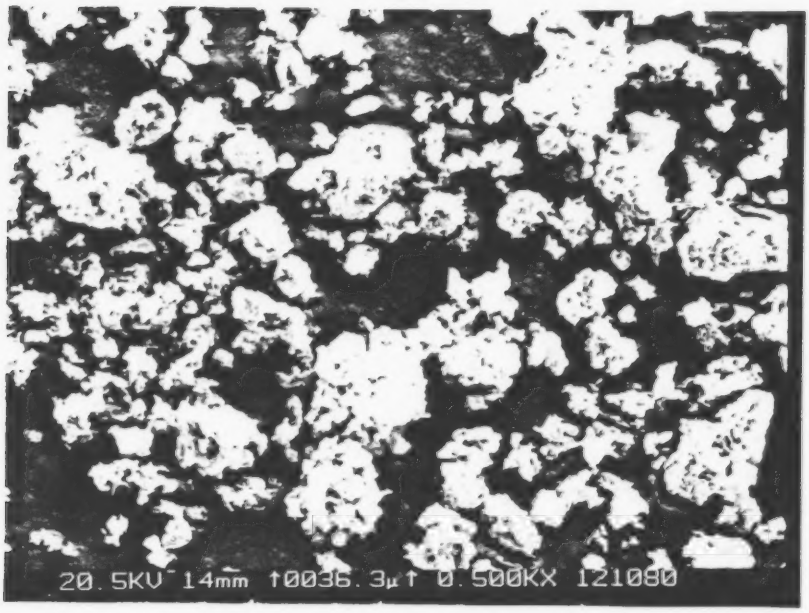
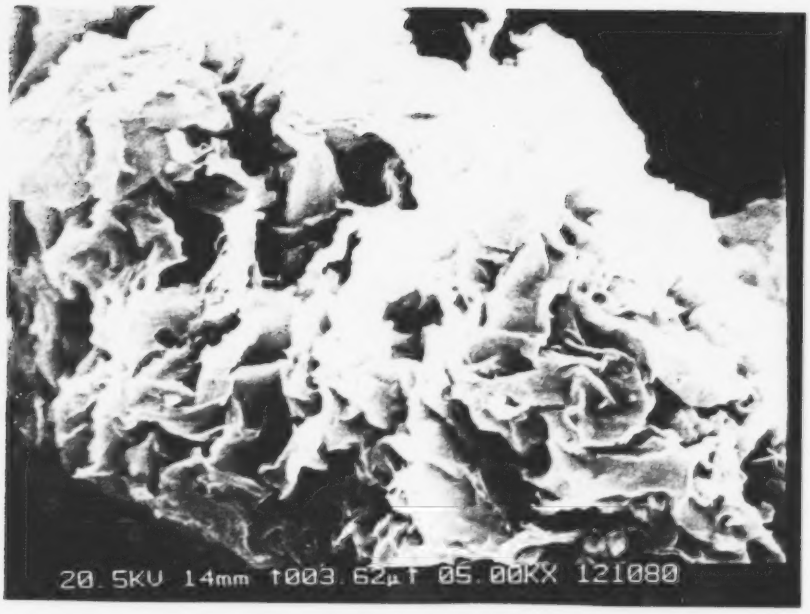
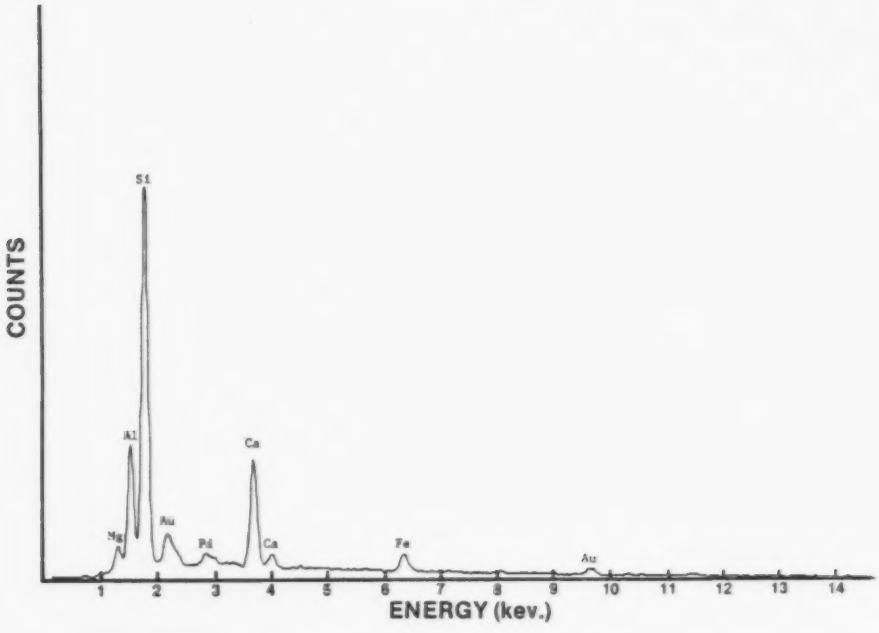


Figure F-34. Scanning Electron Micrograph of Montmorillonite After Thirteen Monthsof Interaction with PFBC Waste Leachate (Magnification = 500X).



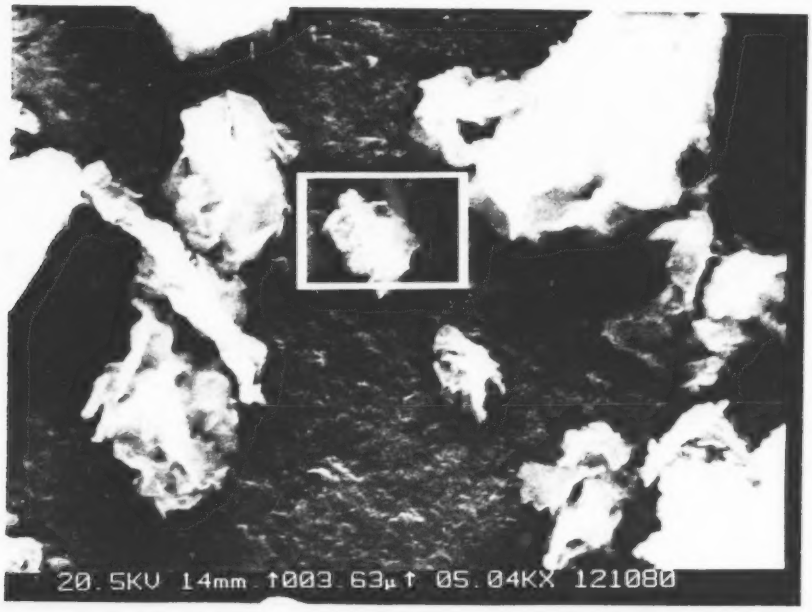
(a.)



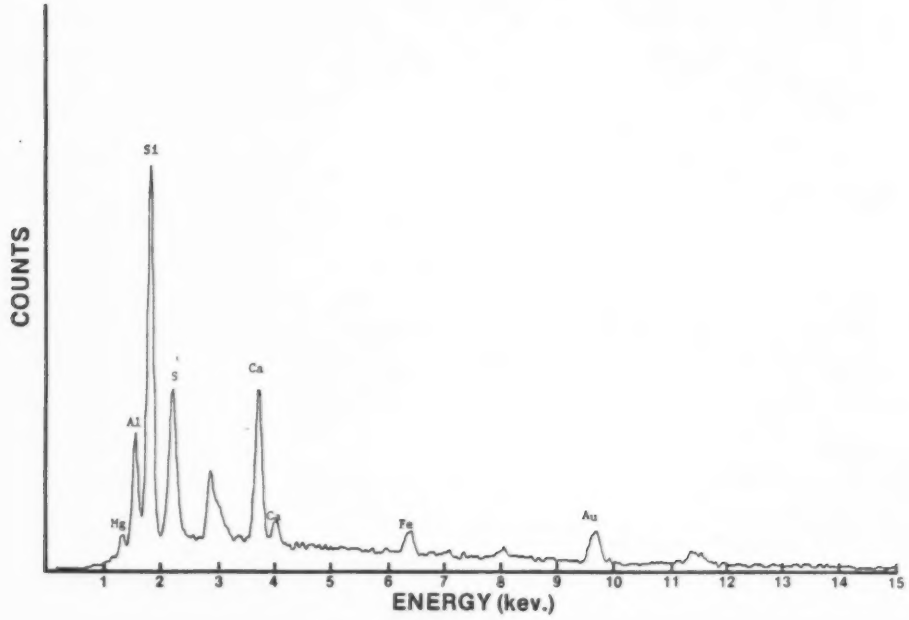
(b.)

Figure F-35. (a.) Scanning Electron Micrograph of Montmorillonite After Thirteen Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).

(b.) X-Ray Fluorescence Spectrum of Chemical Analysis for the Viewed Rosette.



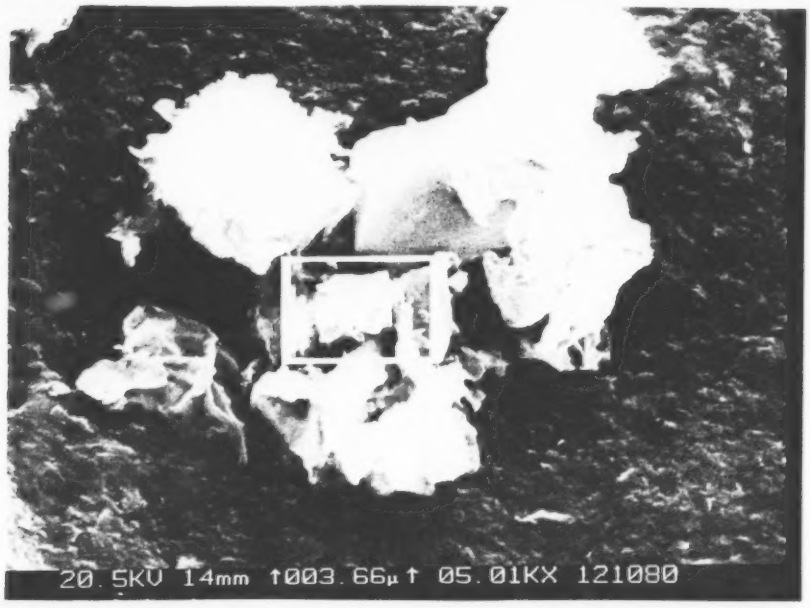
(a.)



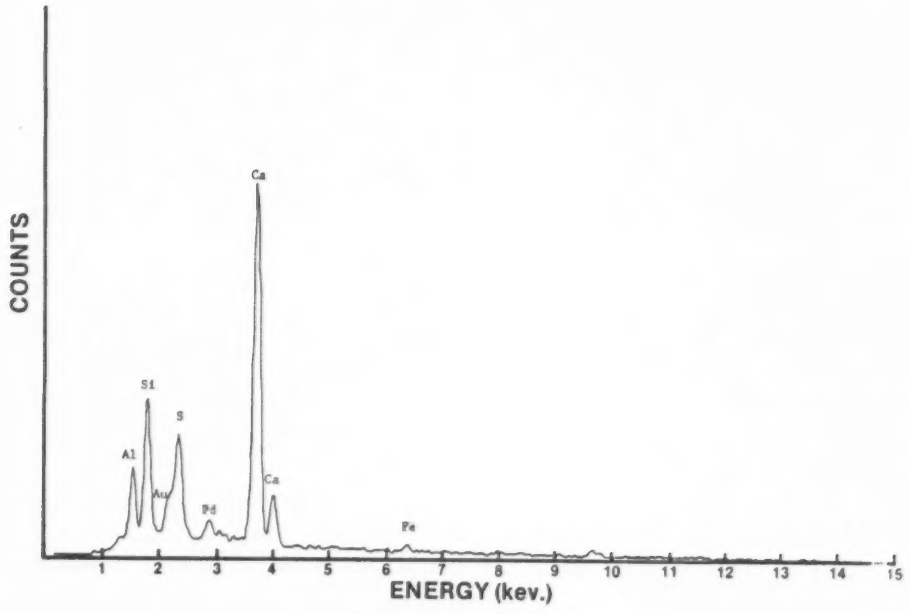
(b.)

Figure F-36. (a.) Scanning Electron Micrograph of Montmorillonite After Thirteen Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).

(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Aggregate.



(a.)



(b.)

Figure F-37. (a.) **Scanning Electron Micrograph of Montmorillonite After Thirteen Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).**

(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Crystal in Framed Area.

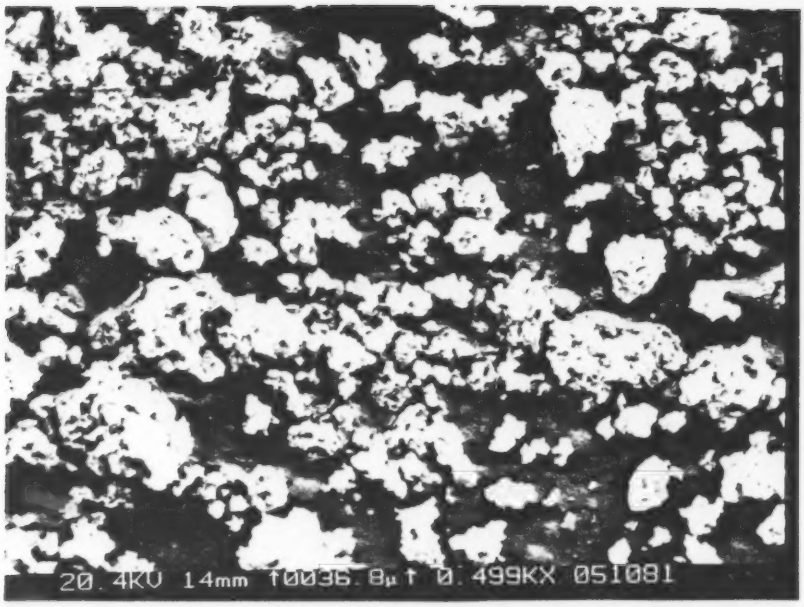
Observations After 18 Months of Interaction

The final clay sample was removed after 18 months of constant agitation in FBC waste leachate. Except for the addition of ettringite and some calcium compounds, the final clay material was unchanged.

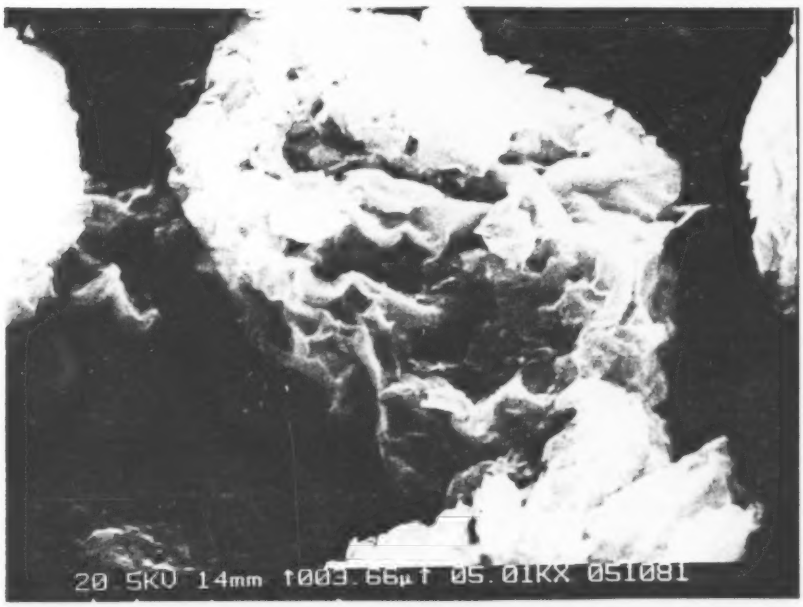
Viewed at low magnification (500X) in Figure F-38, new crystallites are observed between rosettes of montmorillonite. Some of the clay aggregates were a little larger after 18 months of interaction. There was no noticeable change in aggregate size distribution in the clay. The rosettes of clay platelets were still fluffy. This can readily be seen in Figure F-38, which shows the final clay sample at higher magnification. The surface was possibly a little eroded, but generally the clay structure appeared stable after 18 months of interaction. The crystals present at the end of this study are shown with their corresponding XRF scans in Figures F-39 and F-40. The first, depicted in Figure F-39, showed no sulfur present and was probably a calcium aluminosilicate formation. The crystals shown adjacent to this material, framed in Figure F-40, appeared to be calcium sulfate, with low concentrations of aluminum and silicon present.

F.7 Discussion

The primary result of this study is that PFBC waste leachate is not particularly corrosive to clay or synthetic organic landfill liner materials under accelerated leaching conditions. As a result, FBC residue should not pose a severe disposal problem even when liners are required. The secondary implication of the study is that the synthetic liners and the natural clay liner held up equally well with no single candidate liner exhibiting far superior behavior in its interaction with PFBC waste leachate. This allows a high degree of flexibility in liner and disposal site selection for these wastes.

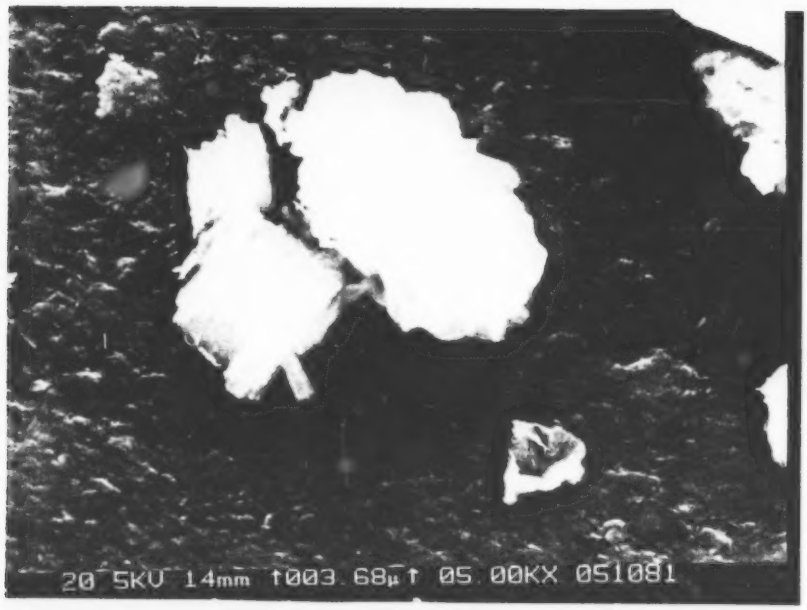


a. Magnification = 500X

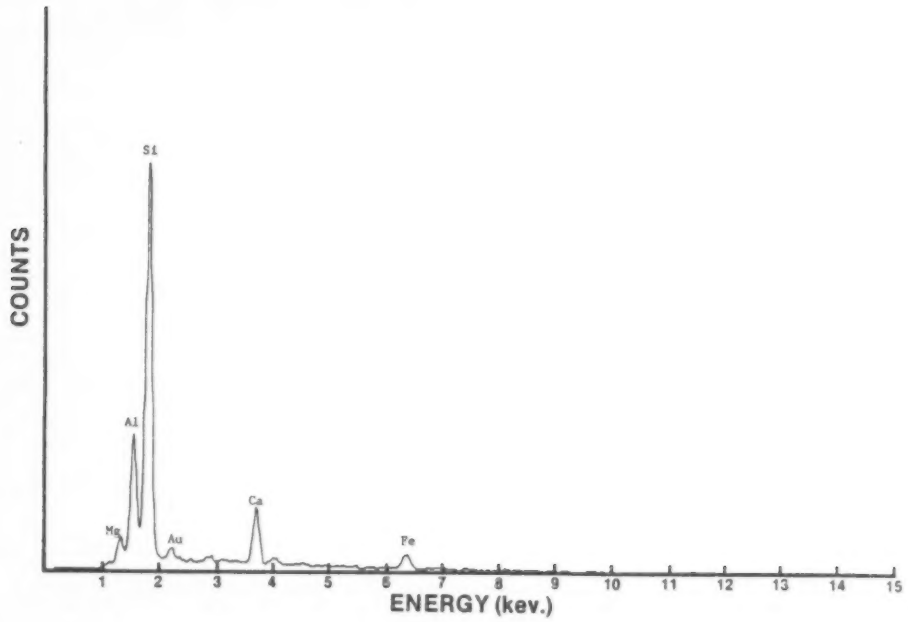


b. Magnification = 5000X

Figure F-38. Scanning Electron Micrograph of Montmorillonite After Eighteen Months of Interaction with PFBC Waste Leachate.

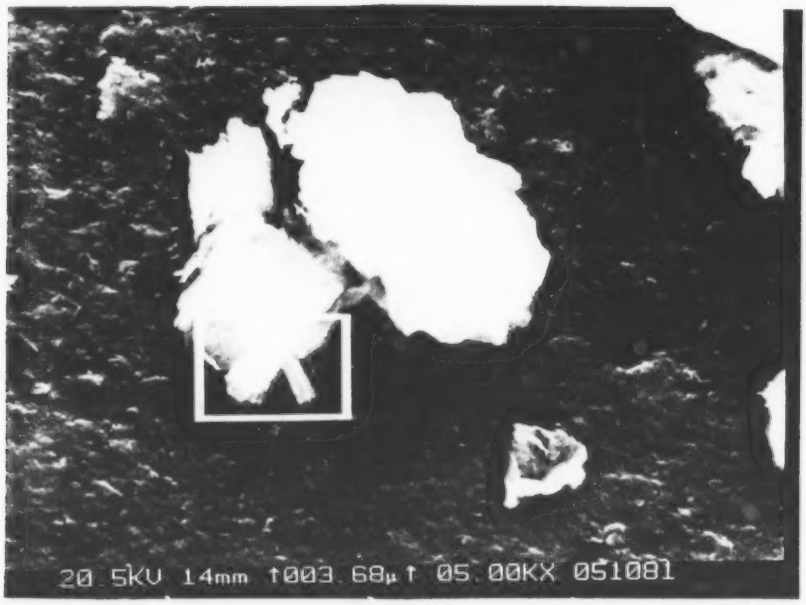


(a.)

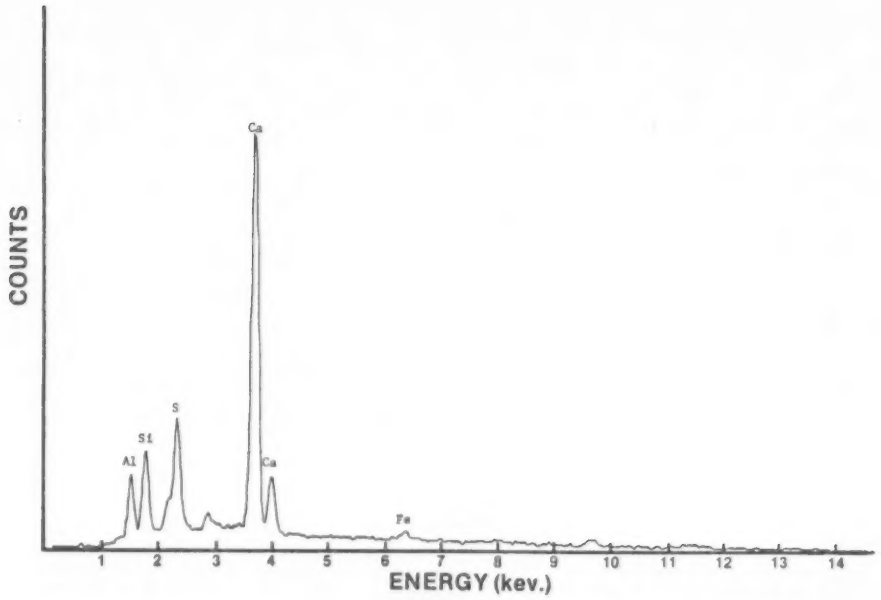


(b.)

Figure F-39. (a.) Scanning Electron Micrograph of Montmorillonite After Eighteen Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).
(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Area.



(a.)



(b.)

Figure F-40. (a.) Scanning Electron Micrograph of Montmorillonite After Eighteen Months of Interaction with PFBC Waste Leachate (Magnification = 5000X).

(b.) X-Ray Fluorescence Spectrum of Chemical Analysis of Framed Crystallite.

F.7.1 Synthetic Liners

Perhaps one of the most confusing implications of these results was that the Hypalon exhibited the optimal tensile strength of the materials studied during leachate interaction. In almost every prior study comparing this material with other landfill liner materials, it was proven to be inferior only because of its low tensile strength (References F-2, F-6, F-7, and F-8). However, the material tested here was heavily reinforced with a mesh of large-gauge fibers deep in the interior of the Hypalon. The tensile strength data for this particular Hypalon material demonstrate that in cases where the wastes that are not highly chemically corrosive, the long-term integrity of a polymer liner is extremely dependent on interior physical reinforcement. Therefore, to assess the maximum liner compatibility information for any waste, a wide range of compositions, reinforcement techniques, grades, plasticizers, and chemical treatments should be considered for each candidate liner material to select a tailor-made substance for containment for a large volume of waste, especially if total containment is mandatory. With the exception of results for Hypalon, these tensile strength data do not appear abnormal according to expected values for these materials based on prior studies.

Although the total organic carbon testing would have been extremely helpful in identifying major breakdown of the polymers, the low concentrations found in this study may be as much a result of final surface chemical treatment of the polymer with plasticizers as it is a function of degration of the polymer itself. The maximum total loss of TOC under these accelerated leaching conditions over the eighteen-month test period represented less than a 5% total loss on a mass basis. More sophisticated analyses would be required to determine if these TOC concentrations represented degradation products of final plasticizer coatings rather than of the polymer itself. It should be noted, however, that TOC concentrations did provide the information required in this study; that is, the low concentrations indicated that the polymer materials were not rapidly decomposing as a result of exposure to the leachate.

One of the major problems encountered in the synthetic polymer study was in the SEM work performed with the polymers that had been exposed to leachate. These polymers developed a fine crystal coating due to the high concentrations of calcium in the FBC waste leachate. Because there was no agitation or flow through the system, calcium compounds slowly precipitated with time. As the SEM samples were removed and allowed to dry, leachate which had diffused to the interior of the materials and which adhered to the surface led to further encrustation of the polymer surface. This crystallization on the surface prevented visual observation of the underlying polymer surfaces with SEM. A mild acid wash of the surfaces was considered along with ultrasonification in warm water and soaking in cold water to reveal polymer surface degradation. However, it was determined that these measures may cause surface effects that are due to sample preparation rather than to long-term interaction with PFBC waste leachate.

Whether a system that incorporates agitation or flow through the immersion tanks would have experienced this precipitation problem is not known. The objective is, of course, to allow surface observation without disturbing the leached polymer surface. Other liner immersion studies have employed flow-through reactors as well as a water-flushing period to remove adherent liquor from the samples prior to their removal for physical testing. Obviously, some method of SEM sample preparation other than the gentle rinsing with deionized water applied to samples in this study is required if the actual polymer surfaces are to be observed by SEM after interaction. Since the data from the polymer strength testing did not indicate major deterioration, the SEM studies may have yielded some informative microscopic surface changes had the surfaces not been coated with precipitate.

F.7.2 Clay Liner

The results from the clay liner investigation were fairly straightforward. Montmorillonite, which is commonly susceptible to attack by ionic salts, was not appreciably affected by constant exposure to FBC leachate under accelerated conditions. Structural and chemical integrity of the montmorillonite lattice during leaching interaction were indicated by the

absence of change in the aluminum concentration in the leachate, the X-ray diffraction patterns, and the SEM observations.

Perhaps the most pressing unanswered question in this part of the study was the long-term effect of ettringite formation from the PFBC leachate and/or clay on the performance of the clay liner. Ettringite, a complex silicate postulated in some studies to be responsible for strength development (and strength loss) in Portland cement mixtures (Reference F-11) may cause brittleness to develop in the clay liner. Whether it is more advantageous to develop a pozzolanic clay mixture or retain the plasticity of a clay material to avoid cracks and resultant seepage is undetermined. It is also not known whether the ettringite is formed solely from the PFBC leachate (inasmuch as it was also found to be present in the leached FBC waste alone, see Text, Section 4.0) or whether ettringite will be formed within the clay liner layer. Whether ettringite forms in the FBC waste or within the clay liner layer would probably affect the clay liner performance.

The SEM observations of the clay liner were much more beneficial in clay liner integrity assessment than in the polymer study. These photomicropraphs provided excellent visual information of the retention of the original clay morphology as well as reinforcement of crystallite formation. The XRF was also invaluable in determining the elemental compositions of the crystal formations.

F.7.3 Test Design

The batch immersion test for liner compatibility assessment is a popular design in current studies (Reference F-8). This test provides a simple, cost-effective design for accelerated leaching of candidate liners. The design used in this study is easy to maintain and use, but it does have drawbacks. The results obtained from a batch immersion test only provide a partial assessment of the liner compatibility.

Column testing should be considered in conjunction with immersion testing (Reference F-8). When liners are placed at the bottom on confined columns containing the waste, laboratory conditions more closely simulate actual field conditions, such as weathering and wet/dry cycles. The column arrangement also provides information concerning the possible interaction of the waste solids as well as the liquid contacting the liner under (artificial) head similar to what may be expected to occur in field conditions.

An important physical parameter that is not measurable in the batch leaching system is permeability. The permeability of both synthetic and clay liners can be measured in the column arrangement. The permeability is one of the best physical parameters for direct comparison of the utility of a synthetic liner versus a clay liner. A column arrangement can also be used to evaluate the quantity and quality of leachate actually seeping past the lienr to the ground water.

Finally, a column arrangement can provide additional information about the mechanism of failure of liner materials. Polymer liners can be removed and examined, and cracking or channeling through a clay layer can be observed.

Other testing not done in this study that might provide valuable information includes laboratory testing of puncture resistance of polymers, assessment of the polymer seam integrity, monitoring of dimensional changes of material, more complete chemical analyses of the materials, and polymer hardness determination. For the clay liners, the cation exchange capacity, conductivity, pH, and permeability could be monitored.

Laboratory conditions more accurately simulating field and actual field studies with longer testing periods would provide better information on liner integrity. The effects of accelerated leaching determined by the technique used in this study cannot be directly correlated to predict degradation on a real-time basis or the effects of the liner interaction with a waste for perpetuity.

F.8 References

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Appendix F

Attachment 1

Tensile Strength Data for the Synthetic Liner Study

A major component of the investigation of the chemical compatibility of six synthetic liner materials with FBC waste leachate was the testing of the tensile strength of the liners before exposure to leachate and again periodically after exposure to the leachate. The raw data for the results are contained in the tables that follow.

TENSILE STRENGTH DATA FOR NEOPRENE

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb.)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	92	81	85	86	6	1,370	8 1/2	7 7/8	7 15/16	8.1	4	810
2	81	97	93	90	9	1,430	7 3/4	9 1/4	9 5/16	9.0	12	900
3	84	91	94	90	6	1,420	8 9/16	9 3/8	9 5/8	9.2	6	920
4	90	86	86	87	3	1,390	8 13/16	8 1/2	8 3/8	8.6	3	860
5	86	97	88	90	6	1,430	8 3/16	9 1/4	8 3/16	8.6	4	860
6	89	85	90	88	3	1,400	8 7/8	8 1/16	8 1/4	8.4	5	840
7	85	92	94	90	5	1,430	7 15/16	8 9/16	8 5/8	8.4	5	840
8	72	67	67	69	4	1,090	7 1/4	5 3/4	5 3/4	6.3	14	630
9	65	67	69	67	3	1,060	5 7/8	6	5 11/16	5.9	3	590
10	60	68	65	64	6	1,020	5 1/8	6	5 3/4	5.6	7	560
11	68	71	69	69	2	1,100	5 15/16	5 7/8	6 1/8	6.0	2	600
12	69	66	68	68	2	1,070	6 1/4	6 1/4	6 7/16	6.3	2	630
13	67	65	65	66	2	1,040	6 5/16	5 15/16	6 1/8	6.1	7	610
14	70	68	65	68	4	1,070	6 1/8	6	6	6.0	1	600
15	72	72	61	68	9	1,080	7 1/4	6 13/16	6	6.7	6	670
16	55	52	55	54	3	860	6 1/4	5 5/8	6 3/8	6.1	7	610
17	53	52	54	53	2	840	5 3/4	6 1/8	5 1/2	5.8	5	580
18	60	60	55	58	5	930	6	5 7/8	5 5/8	5.8	3	580
initially (pre-leaching)	91	90	87	89	2	1,420	8	8	8	8.2	4	720

* Cross sectional area of strip = 1" x 0.063" = 0.063 in².

TENSILE STRENGTH DATA FOR POLYVINYL CHLORIDE

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb.)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	62	62	63	62	1	1,940	6 11/16	6 1/2	6 7/8	6.7	3	670
2	56	62	64	61	7	1,910	6 3/8	6 1/2	6 3/4	6.5	3	650
3	63	63	61	62	2	1,940	6 5/8	6 7/8	6 13/16	6.8	2	680
4	59	62	62	61	3	1,910	6	6 13/16	6 11/16	6.5	7	650
5	63	67	70	67	5	2,090	6 3/4	7 1/4	7 5/16	7.1	4	710
6	70	65	66	67	4	2,090	7 1/2	7 1/8	7 3/16	7.3	3	730
7	66	64	64	65	2	2,030	7 3/16	7 1/16	7 3/8	7.2	2	720
8	57	52	50	53	7	1,660	5 5/16	4 1/2	4 3/16	5.0	23	500
9	56	52	51	53	5	1,660	5 1/2	4 5/8	4 1/4	4.8	15	480
10	50	46	53	50	5	1,560	4 1/4	4 7/16	3 3/4	4.1	9	410
11	50	53	48	50	5	1,560	4 3/8	4 13/16	4 3/16	4.5	7	450
12	58	56	52	55	6	1,720	5 15/16	5 3/4	5 5/16	5.7	6	570
13	48	48	46	47	2	1,470	3 7/8	4 7/16	4 1/16	4.1	7	410
14	55	56	55	55	1	1,720	5 1/16	5 11/16	5 1/4	5.3	6	530
15	52	53	53	53	1	1,660	5 5/16	5 3/8	5 1/4	5.3	1	530
16	45	42	41	43	5	1,340	5 1/2	5	4 7/8	5.1	6	510
17	44	36	45	42	12	1,300	5	3 5/8	5	4.5	18	450
18	47	48	47	47	1	1,480	5 3/4	5 3/4	5 13/16	5.8	1	580
initially (pre-leaching)	66	50	40	52	25	1,625	8	5	3 5/8	5.5	40	550

* Cross sectional area of strip = 1" x 0.032" - 0.032 in².

TENSILE STRENGTH DATA FOR CHLORINATED POLYETHYLENE

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	45	45	51	47	7	1,570	12	12	12 15/16	12.3	4	1,230
2	50	49	51	50	2	1,670	12 13/16	11 1/4	11 5/8	11.9	7	1,190
3	50	50	50	50	0	1,670	11 7/16	11 1/4	11	11.2	2	1,120
4	49	47	47	48	2	1,600	10 7/8	10 7/8	10 3/4	10.8	1	1,080
5	46	42	46	45	2	1,500	10 9/16	9 7/8	10 3/4	10.4	4	1,040
6	46	45	44	45	2	1,500	10 3/4	10 5/8	10 1/2	10.6	1	1,060
7	38	39	41	39	4	1,300	8 1/4	8 7/8	9 13/16	9.0	9	900
8	40	36	39	38	5	1,270	8 3/4	7 11/16	8 9/16	8.3	7	830
9	36	35	33	36	5	1,200	8	8	7 7/8	8.0	1	800
10	36	40	31	36	13	1,200	7 1/2	9 1/4	6 7/8	7.9	16	790
11	37	40	23	33	27	1,100	8 1/8	9 1/8	5 3/4	7.7	23	770
12	30	33	37	33	11	1,100	7 3/8	8 1/8	9 1/8	8.2	11	820
13	21	35	32	29	25	970	5 1/2	8 1/2	8 7/16	7.5	23	750
14	30	33	24	29	16	970	6 1/4	7 5/16	5 5/8	6.4	13	640
15	19	18	19	19	3	630	3 5/8	3 1/2	3 9/16	3.6	2	360
16	29	23	25	26	12	870	7 7/8	6	6 7/8	6.9	14	690
17	29	31	29	30	4	990	7 3/4	8	7 5/8	7.8	2	780
18	27	24	27	26	7	870	5 3/4	5 1/4	5 3/4	5.6	5	560
initially (pre-leaching)	40	42	46	43	7	1,430	10 1/4	10 11/16	11 3/16	8.8	25	880

* Cross sectional area of strip = 1" x 0.03" = 0.03 in².

TENSILE STRENGTH DATA FOR HYPALON

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb.)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	≥100	≥100	≥100	≥100	0	≥2,170	1	1 3/16	1	1.1	11	110
2	≥100	92	≥100	≥97	5	≥2,110	0 13/16	1	0 13/16	0.9	12	90
3	≥100	≥100	≥100	≥100	0	≥2,170	1 1/4	0 7/8	1 1/8	1.1	18	110
4	≥100	≥100	≥100	≥100	0	≥2,170	0 3/4	0 15/16	0 7/8	0.9	11	90
5	≥100	≥100	≥100	≥100	0	≥2,170	1 5/16	1	0 7/8	0.9	10	90
6	≥100	≥100	≥100	≥100	0	≥2,170	0 15/16	1 1/16	0 7/8	1.0	10	100
7	≥100	88	89	92	7	≥2,000	1 5/16	1 3/8	1 1/2	1.4	7	140
8	72	92	99	88	16	1,910	3 1/16	3 3/4	2 5/8	3.1	18	310
9	88	≥100	98	95	7	≥2,070	2	1 9/16	2 1/8	1.9	16	190
10	91	94	≥100	95	5	≥2,070	1 7/8	2 1/4	2 7/16	2.2	13	220
11	90	92	99	94	5	2,040	3 1/16	2 1/8	2 5/16	2.5	20	250
12	94	≥100	≥100	98	4	≥2,130	2 3/8	2 5/8	2 3/8	2.4	6	240
13	93	≥100	≥100	98	4	≥2,130	2 1/8	2 1/4	2 7/16	2.3	7	230
14	76	82	74	77	5	1,670	3 1/8	3 3/16	3	3.1	3	310
15	83	82	82	82	1	1,780	2 5/8	3 5/16	2 7/16	2.8	36	280
16	84	83	89	85	4	1,850	2 11/16	2 1/16	2 1/4	2.3	14	230
17	87	80	85	84	4	1,830	2 3/4	3 3/8	3 1/4	3.1	11	310
18	73	67	80	73	9	1,590	2 5/8	3 3/8	3 1/2	3.2	15	320
initially (pre-leaching)	≥100	≥100	≥95	≥98	3	≥2,130	1 7/8	2 1/8	2	2	6	200

* Cross sectional area of strip = 1" x 0.046" = 0.046 in².

TENSILE STRENGTH DATA FOR BUTYL RUBBER

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb.)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	78	65	80	74	11	1,300	12	7 11/16	12	10.6	24	1,060
2	83	79	92	85	8	1,490	12	12	12	12.0	0	1,200
3	92	93	92	92	1	1,610	11 3/16	10 3/4	12	11.3	6	1,130
4	92	95	94	93	1	1,630	11 1/2	11 1/2	11 7/8	11.6	2	1,160
5	92	93	94	93	1	1,630	11 5/8	12	11 9/16	11.7	2	1,170
6	92	70	93	85	15	1,490	12	8 1/4	11 7/8	10.7	20	1,070
7	94	80	83	86	9	1,510	12	10 1/16	10 1/8	10.7	10	1,070
8	71	65	55	64	13	1,120	9 9/16	7 1/8	5 5/8	7.4	27	740
9	56	73	57	62	15	1,090	5 3/4	9 1/4	5 5/8	6.9	30	690
10	67	63	65	65	3	1,140	7 1/16	7 3/16	6 11/16	7.0	4	700
11	70	65	74	70	6	1,230	8 5/8	6 3/4	8 3/4	8.0	14	800
12	59	57	57	58	2	1,020	5 9/16	5 7/16	5 5/16	6.5	26	650
13	63	53	71	62	14	1,090	6 7/8	4 13/16	8 3/8	6.7	27	670
14	71	55	65	64	13	1,120	7 3/4	4 13/16	6 5/8	6.4	23	640
15	75	73	69	72	4	1,260	8 7/8	8 15/16	9	8.9	1	890
16	60	62	60	61	2	1,070	8 3/4	9 1/2	8 9/16	8.9	6	890
17	57	60	57	58	3	1,020	10 3/8	8 7/8	10 1/4	9.8	8	980
18	64	57	63	61	6	1,080	9 1/2	7 1/4	11 1/4	9.3	22	930
initially (pre-leaching)	80	79	71	77	6	1,350	12	9	7 7/8	9.6	22	960

* Cross sectional area of strip = 1" x 0.057" = 0.057 in².

TENSILE STRENGTH DATA FOR ETHYLENE PROPYLENE DIENE RUBBER

Leaching Exposure Time (months)	Applied Force at Ultimate Elongation (lb.)					Tensile Strength* (lb/in²)	Distance Stretched to Rupture (in.)					Ultimate Elongation (%)
	Break 1	Break 2	Break 3	Mean	Standard Deviation (%)		Length 1	Length 2	Length 3	Mean	Standard Deviation (%)	
1	99	≥100	≥100	≥100	1	≥1,540	7 7/8	7 11/16	7 3/16	7.6	5	760
2	≥100	97	≥100	≥99	2	≥1,520	7 3/4	7 1/2	8	7.8	3	780
3	≥100	≥100	≥100	≥100	0	≥1,540	7 7/8	7 5/8	7 7/8	7.8	2	780
4	≥100	≥100	≥100	≥100	0	≥1,540	8 3/8	8 1/8	7 15/16	8.1	3	810
5	99	100	100	100	1	1,540	8 1/4	8 3/8	8 1/16	8.2	2	820
6	100	100	96	99	2	1,520	8	7 13/16	7 15/16	7.9	1	790
7	≥100	≥100	97	99	2	1,520	8 5/16	12	7 3/4	9.3	25	930
8	85	77	74	79	7	1,260	7 3/8	6 13/16	6 3/8	6.9	7	690
9	79	75	74	76	3	1,170	6 7/8	6 3/8	6 9/16	6.6	4	660
10	74	77	76	76	2	1,170	6 9/16	7 1/8	7	6.9	4	690
11	81	81	86	83	3	1,270	7 1/2	7 5/16	8 3/16	7.7	6	770
12	74	80	84	79	6	1,220	7 1/8	7 1/2	7 7/8	7.5	5	750
13	82	79	84	82	3	1,260	7 7/16	7 1/2	7 7/8	7.6	3	760
14	82	88	80	83	5	1,270	7 13/16	8 1/16	7 9/16	7.7	3	770
15	81	84	79	81	3	1,250	7 1/4	7 1/2	7 1/16	7.3	3	730
16	75	68	70	71	5	1,090	7 5/16	6 3/4	7	7.0	4	700
17	71	74	70	72	3	1,100	7 1/4	7 3/4	7 1/4	7.4	4	740
18	65	68	70	68	4	1,040	6 1/2	7	7 1/8	7.1	7	690
initially (pre-leaching)	≥100	≥100	≥100	≥100	0	≥1,540	8 11/16	8 5/8	8 7/8	8.7	1	870

* Cross sectional area of strip = 0.065 in².